

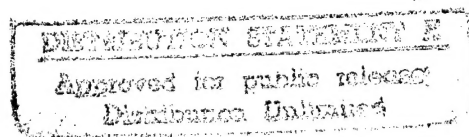
HYBRIDIZED POLYMER MATRIX COMPOSITES

Bruce A. Stern
Teunis Visser

Prepared for
NASA-LEWIS RESEARCH CENTER
Cleveland, Ohio

19960205 024

RECEIVED
LEWIS RESEARCH CENTER
SEP 15 1981
September 1981



DMIC QUALITY INSPECTED 1



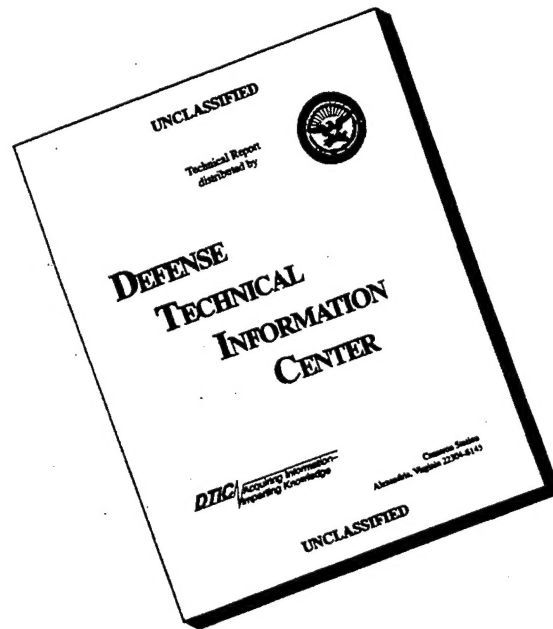
**COMPOSITES
HORIZONS, INC.**

2303 WEST VALLEY BOULEVARD • POMONA, CALIFORNIA 91768

(714) 623-6902

4076

DISCLAIMER NOTICE



THIS DOCUMENT IS BEST QUALITY AVAILABLE. THE COPY FURNISHED TO DTIC CONTAINED A SIGNIFICANT NUMBER OF PAGES WHICH DO NOT REPRODUCE LEGIBLY.

1. Report No. NASA CR-165340		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle HYBRIDIZED POLYMER MATRIX COMPOSITES				5. Report Date September 1, 1981	
				6. Performing Organization Code	
7. Author(s) Bruce A. Stern and Teunis Visser				8. Performing Organization Report No.	
				10. Work Unit No.	
9. Performing Organization Name and Address Composites Horizons 2303 W. Valley Blvd. Pomona, CA 91768				11. Contract or Grant No. NAS3-21384	
				13. Type of Report and Period Covered Contractor Report - Final	
12. Sponsoring Agency Name and Address NATIONAL AERONAUTICS AND SPACE ADMINISTRATION WASHINGTON D.C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes PROJECT MANAGER, T. T. SERAFINI, MATERIALS DIVISION NASA-LEWIS RESEARCH CENTER, CLEVELAND, OHIO 44135					
16. Abstract Under certain conditions of combined fire and impact, graphite fibers can possibly be released to the atmosphere by graphite fiber composites. This program was conducted to improve the retention of graphite fiber in these situations. Hybrid combinations of graphite tape and cloth, glass cloth, and resin additives were studied with epoxy and polyimide resin systems. Polyimide resins formed the most resistant composites and resins based on simple novolac epoxies the least resistant of those tested. Great improvement in the containment of the fibers was obtained using graphite/glass hybrids, and nearly complete prevention of individual fiber release was made possible by the use of resin additives.					
17. Key Words (Suggested by Author(s)) Graphite reinforced composites Hybrid composites Epoxy, polyimide, epoxy resins Mechanical properties Flammability testing Impact testing				18. Distribution Statement Unclassified, unlimited	
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages	
				22. Price*	

FOREWORD

This final technical report covers the work performed under NASA Contract NAS3-21384 "Hybridized Polymer Matrix Composites". The program was sponsored by NASA Lewis Research Center, Cleveland, Ohio. Dr. Tito T. Serafini was the NASA Project Manager.

At Composites Horizons the program was directed by Bruce A. Stern, Program Manager. Teunis Visser, acting as Project Engineer, was the Principal Investigator. Thomas Crawford assisted in the mechanical and physical testing. Dorothy West and Andrea Petker assisted in panel fabrication.

TABLE OF CONTENTS

	PAGE
SUMMARY	1
1.0 INTRODUCTION	2
2.0 TECHNICAL DISCUSSION	3
2.1 CONCEPT SELECTION AND ANALYSIS	3
2.1.1 SELECTION CRITERIA	4
2.1.2 SELECTION CONCEPTS	5
2.1.3 LAMINATE SCREENING STUDY	8
2.1.4 ADDITIVE SCREENING STUDY	14
2.1.5 LAMINATE ANALYTICAL STUDY	18
2.2 FABRICATION AND EVALUATION OF SELECTED CONCEPTS	19
2.2.1 LAMINATE FABRICATION	19
2.2.2 FLAMMABILITY/IMPACT TESTING	21
2.2.3 MECHANICAL/PHYSICAL PROPERTY EVALUATION	22
2.2.4 FINAL CONCEPT SELECTION	24
3.0 CONCLUSIONS	24
REFERENCES	26

LIST OF FIGURES

FIGURE		PAGE
1.	HYBRIDIZING CONCEPT SURFACE PLIES	27
2.	HYBRIDIZING CONCEPT CENTRAL CORE SECONDARY REINF. AND SURFACE PLIES	27
3.	HYBRIDIZING CONCEPT FULL INTERLAMINAR MIXING OF PRIMARY AND SECONDARY REINF.	27
4.	SCHEMATIC OF BURN IMPACT TESTER	28
5.	CALIBRATION CURVE IMPACT ENERGY VS. CYLINDER PRESSURE	29
6.	PHOTO CC11 TRANSVERSE FIBERS 1 X 4 SPECIMENS	30
7.	CC11 LONGITUDINAL FIBERS 1 X 4 SPECIMENS	31
8.	CC11 3 X 4 SPECIMEN	31
9.	FIBERS ON AIR FILTER AND BOTTOM OF TESTER FROM TEST OF SPECIMEN FROM FIGURE 7.	32
10.	UNPROTECTED GRAPHITE PANEL TEST.	33
11.	KYNOL FACED PANEL TEST.	33
12.	PMR15 UD PANEL TEST	34
13.	DL 23 BURN/IMPACT TEST	35
14.	FIBERS ON AIR FILTER FROM TEST OF SPECIMEN FROM FIGURE 12.	35
15.	FACING VIEW CD5 FOLLOWING BURN/IMPACT TEST	36
16.	SIDE VIEW CD5 FOLLOWING BURN/IMPACT TEST	36
17.	EJECTA FROM BOTTOM OF TESTER CD5 TEST	37
18.	TEST RESULTS OF CH-8 SAMPLE	38
19.	SAMPLE FROM CONFIGURATION DL-18	39
20.	TEST OF CC34	39
21.	SAMPLE FROM DL-27	40

FIGURES (CONT.)

FIGURE		PAGE
22.	TEST RESULTS, CH10	41
23.	SAMPLE FROM CH5	42
24.	SAMPLE FROM CD24	43
25.	SAMPLE FROM CD21	44
26.	SAMPLE FROM CH16	45
27.	SIDE VIEW AFTER BURN/IMPACT TESTING OF DL-9	46
28.	EJECTA FROM BOTTOM OF TESTER, DL 23 TEST	46
29.	SAMPLE FROM CD-6	47
30.	PANEL DL-3 AFTER BURN/IMPACT TESTING	48
31.	EJECTA FROM BOTTOM OF TESTER, DL-3 TEST	49
32.	ULTRASONIC C SCAN RESULTS, 4 PANELS	50
33.	PHOTOMICROGRAPH, PANEL CH-8	51
34.	PHOTOMICROGRAPH, PANEL DL-18	51
35.	PHOTOMICROGRAPHS OF CC34 SAMPLES	52
36.	PHOTOMICROGRAPH, PANEL CH-13	53
37.	PHOTOMICROGRAPH, PANEL CC-11	53
38.	PHOTOMICROGRAPH, PANEL DL-27	54
39.	PHOTOMICROGRAPH, PANEL CC-18	54
40.	PHOTOMICROGRAPH, PANEL CH-10	55
41.	PHOTOMICROGRAPH, PANEL CH-5	56
42.	PHOTOMICROGRAPH, PANEL CD-24	56
43.	PHOTOMICROGRAPH, PANEL CD-21	57
44.	PHOTOMICROGRAPH, PANEL CD-16	57
45.	PHOTOMICROGRAPH, PANEL DL-9	58

LIST OF TABLES

TABLE		PAGE
1.	SELECTION CRITERIA	59
2.	MATERIALS AND IDENTIFICATION	60
3.	BURN IMPACT TESTER COMPONENTS	61
4.	CONFIGURATION AND CALCULATED THICKNESS, THIN PANELS	62
5.	CONFIGURATION AND CALCULATED THICKNESS, THICK PANELS	63
6.	BURN IMPACT DATA WITH AND WITHOUT ADDITIVE	64
7.	MECHANICAL TEST WITH AND WITHOUT ADDITIVE	64
8.	CALCULATED PROPERTIES THIN PANELS	65
9.	CALCULATED PROPERTIES THICK PANELS	66
10.	BURN/IMPACT TEST RESULTS, THIN PANELS	67
11.	BURN/IMPACT TEST RESULTS, THICK PANELS	68
12.	ACID DIGESTION RESULTS, HYBRID PANELS	74
13.	SHORT BEAM SHEAR STRENGTH, HYBRID PANELS	75
14.	FLEXURAL STRENGTH, HYBRID PANELS	76
15.	FLEXURAL MODULUS, HYBRID PANELS	77
16.	SELECTED CONFIGURATIONS FOR DELIVERED PANELS	78

SUMMARY

This report describes work done to improve the retention of graphite fiber by graphite fiber reinforced composite material under conditions of fire and impact exposure. The approach investigated in this program was the "hybridization" of the composite. As used in this study, the term "hybrid" means the use of additional materials other than the graphite fiber and the matrix resin to change the graphite fiber retention characteristics of the resultant hybrid composite. A major constraint in this program was to utilize as a baseline, materials that were already being widely used as composites to which improvements in fiber retention were to be made. Based on the impact tests performed in the program, the use of other fiber reinforcements such as glass, and the use of resin additives were found to provide improved graphite fiber retention. The use of glass cloth/graphite fiber hybrid composites offers an effective, immediate, and practical approach to improving fiber retention. The resin additives uncovered in this program offer an even greater potential for improvement in this area, but further studies are required to fully characterize the effect of these additives on the mechanical behavior of the resulting composite.

1.0 INTRODUCTION

It can be expected that in the future there will be a proliferation of graphite fiber based composites in aircraft and other transportation system structures, and that at some point these parts will be subjected to fire and impact conditions. Concern was expressed that these fires would release large quantities of fibers into the atmosphere (Ref 1). Graphite fibers being very small in diameter, light in weight, and very conductive electrically were considered to pose a special risk in that they could be dispersed very readily and might cause damage to electrical devices. A specific chain of events would be required for this to occur, involving a number of distinct steps. (1) There is a destructive fire that involves a large quantity of graphite composite. (2) The fire vaporizes, burns, or pyrolyzes the organic resin from the composite. (3) A mechanical disturbance (e.g. crash) breaks and shortens the long graphite fibers into smaller, separated, and easily movable pieces. (4) Air currents carry these very light fibers away from the burn site. (5) The fibers penetrate into an electrical apparatus. (6) The fibers bridge conductors in the apparatus causing short circuits.

As a result of the concern over this potential problem, NASA funded several risk analysis and materials programs to investigate various aspects of this subject (Ref.2). In general it was concluded

that the danger of real damage from the uncontrolled release of graphite fiber is small. This program under NASA contract number NAS3-21384 had the objective of improving the retention of graphite fiber in fire/impact situations by hybridizing the composite structure. Combinations of materials were investigated to achieve the desired behavior of the composite based on selection criteria that would permit the use of these materials in actual composite structure.

2.0 TECHNICAL DISCUSSION

This program was divided into two major technical tasks. The first task included the selection analysis and screen testing of baseline and hybrid laminate concepts, and of resin additives. The second task was the fabrication and testing of laminates of the selected concepts. This task culminated in the selection, fabrication and delivery to NASA of laminates representing the best of the concepts evaluated.

2.1 CONCEPT SELECTION AND ANALYSIS

The first phase of this program was the selection of hybridization concepts, an analysis of their properties and two series of screening studies. The first series was devoted to burn/impact trials of baseline and trial laminate concepts, and the second was devoted to resin additives.

2.1.1 SELECTION CRITERIA

To provide the basis for maximizing the utility of any hybridizing and materials concepts developed, certain selection criteria were imposed on the candidate concepts. These are summarized in Table 1. The ability to translate a successful concept into actual structural use meant that certain conditions must be met. The selected material must be processible, cost effective, and not result in a weight penalty that would preclude its use. Additionally, the program targeted composites in two basic thickness ranges, 0.64 - 1.02 mm (0.025-0.040 inch) and greater than 6.4 mm (0.25 inch). These represent the extremes of typical structural composite use, from thin sandwich skins to heavier structural elements. Due to their widespread use in aircraft structure, primarily epoxy resins were used in the hybrid evaluations. Some tests were performed on PMR15 polyimide composites to establish the influence of a more thermally capable resin on the fire/impact behavior of the composite.

To address the weight consideration, an arbitrary lower modulus limit of approximately 6.9×10^3 Mpa (10 Msi) was set for concepts basically comprised of unidirectional graphite fiber. In these cases, the transverse reinforcement was provided by the hybridizing fiber, usually glass. At this level of tensile modulus, there is still a weight advantage over aluminum, based on relative densities of aluminum and the composite. Cross-plyed graphite composites were considered without this constraint because these composites would likely be used as shear webs or skins requiring shear and/or torsional

rigidity. The determination of modulus was made through use of the computer program "LAMSTIF1"¹ which utilizes the single ply properties of the constituent materials, their orientation and thickness to calculate the stiffness characteristics of the hybrid composite as a whole. Using this program a wide variety of combinations of materials and stacking sequences were evaluated to arrive at candidate configurations meeting the stiffness conditions desired for the structure. All of the concepts actually considered had ply orientations restricted to 0, ±45, and 90 degrees, and were typically symmetrical about the centerline of laminate thickness. All of these would be capable of being fabricated as easily as current composite structures.

2.1.2 SELECTION CONCEPTS

In approaching the problem of fiber retention two lines of attack were taken. First, the hybridizing of the composite by means of alternate fibers within the same resin matrix was considered; and, secondly, the use of alternative resins and resin additives was considered. The incorporation of alternate fibers was viewed with the idea that these materials could be used to impede the oxidative attack on the composite and/or confine the fibers within the composite by acting as a net once the resin had burned off. The most promising material considered as a secondary reinforcement was fiberglass cloth. In the concepts considered, glass was incorporated as surface material, as interlaminar plies, and mixed intralaminarly in a hybrid glass/graphite cloth.

¹ Program in BASIC written by Bruce A. Stern, Composites Horizons for the TRS80 (TANDY Corp.) microcomputer system.

The use of glass, usually in the cloth form, offered a means of keeping the overall cost of the composite down while minimizing penalties to laminate strength. In primarily unidirectional graphite reinforced composites, glass cloth was utilized to provide transverse stiffness and strength. Concepts were considered with wide varieties of complexity in incorporating the glass both at the surface and within the composite. As determined by the testing done at the screening level and in the final laminates selected, the use of glass made possible dramatic reductions in the quantity of graphite released by a composite after a fire/impact exposure.

Specific design approaches for hybrid composite structures can cover a very wide range of material combinations. Some preconditions immediately eliminate many of the combinations. The first condition is that the graphite fiber form the primary reinforcement. The second is that the resultant structure should offer a performance advantage in cost, weight or both, compared to a similar metallic component. This latter criteria made it desirable to maximize the volume fraction of graphite compared to a lower modulus secondary reinforcement such as fiberglass. A further consideration was that the use of graphite toward the outside of a hybrid could increase its structural advantage, enabling the use of a lower cost reinforcement as the central portion of the laminate. Unfortunately, such a use places the graphite fiber nearer the laminate surface which is potentially a more vulnerable position.

Figures 1, 2 and 3 show schematics of three different interlaminar hybridization concepts. In Figure 1 the hybridization, or secondary, reinforcement is utilized in the outer plies of the structure. Glass cloth offered promise for the outer plies because glass cloth has a relatively low thermal conductivity, and could retain some of its integrity even after the resin matrix had burned away. The type of interlaminar hybridization is shown in Figure 2 utilizes a central core of secondary reinforcement with graphite on either side. Additionally secondary reinforcement forms the outer plies. For some applications this "sandwich" approach could offer a way to utilize a minimum quantity of graphite while using less expensive as secondary reinforcement materials. The full interlaminar mixing of primary and secondary reinforcements is shown in Figure 3. In this case property retention may be adjusted through the use of a variety of thicknesses of secondary reinforcement plies. In this program a range of thicknesses of glass cloth was utilized from 7781 style, at approximately .25 mm (.004 in.) per ply, to 120 style, 0.10 mm (.004 in.) per ply, to 104 style, at 0.03 mm (0.001 in.) per ply. Combinations of these materials were also evaluated in the laminate screening study (see Section 2.1.3).

Intralaminar mixing of reinforcements was also evaluated in this program. A woven graphite/glass cloth hybrid construction was selected that provided nearly unidirectional graphite properties yet had transverse glass tying the graphite tows together. This

cloth (style W190) was obtained from Fiberite. This material was further hybridized in use with additional glass cloth layers using the concepts noted above.

Other fibers were considered and evaluated in the laminate screening study discussed in Section 2.1.3. Among these were a phenol-formaldehyde based cloth (Kynol) and Kevlar cloth. These were considered for use as potential surface plies because they were reputed to be high char yield materials. As the screening test results showed, however, neither material was effective in reducing the release of graphite fibers in the fire/impact testing done in this program. In neither case did the char formed by the burning of these materials adhere to the graphite fibers within the composite.

While the basic resin utilized in this program was an epoxy, some testing was done on graphite fiber composite made with PMR15 polyimide resin to determine the effect of a more thermally resistant resin. Emphasis was placed on resin additives that might in part improve the flame resistance, char yield, or other aspects of the behavior of the composite with a resultant improvement in the retention of graphite fiber. This study is discussed in Section 2.1.4.

2.1.3 LAMINATE SCREENING STUDY

The selection of candidate configurations for the detailed evaluation was based on the screening study of a variety of hybrid

design concepts. The materials employed were ones that were readily available and already in use in graphite composite structures in test or service. Five epoxy resin systems were used in the making of panels. These were Narmco 5208, Hexcel F263, Fiberite 934, Ferro CE9000, and CH4010 (used by Composite Horizons in jet engine hardware fabricated for Pratt & Whitney Aircraft). PMR15 polyimide resin was also included in the screening study. The graphite fiber reinforcement material was used in cloth and tape forms. The tape thickness was typically 0.13 mm (0.005 in.) per ply, and the all graphite cloth was a standard eight harness satin construction (23 X 24), W133 as supplied by Fiberite. The W190 graphite/glass hybrid cloth had a thickness of approximately 0.18 mm (0.007 in.) per ply. All of the graphite used was of the high strength variety. The glass cloth types were described in Section 2.1.2. Kevlar cloth was used in a 281 style weave 0.25 mm (0.010 in.) per ply. Table 2 summarizes the materials used and their identifications as incorporated into subsequent tables and figures.

Following the selection of a variety of concepts using these materials and a computer analysis to verify that the concepts met the selection criteria, the actual screening was done by fabricating test laminates and subjecting them to a burn impact test. The burn/impact test apparatus used was similar to the apparatus described by Richard Fish (Ref. 3). The apparatus allowed panels to be

heated radiantly at temperatures up to 1000°C and was equipped with a device to impact the burned area with a reproducible force, without the need to remove the specimen. Figure 4 provides a schematic of the tester components. A list of these components is provided in Table 3.

Temperatures of the burning panels were monitored by means of an optical pyrometer. Calibration of the impact energy of the "tup" on the air cylinder was first attempted by measuring the speed of the tup at the plane of impact by means of an interrupted laser beam. This calibration did not result in a useful range of impact energy versus cylinder pressure, so an additional calibration was performed by comparing the effect of impacting pure lead castings by means of the tester (at various pressures) and by dead weight drops of the same tup from known heights. The resulting calibration curve of impact energy versus cylinder pressure is presented in Figure 5. To obtain the most destructive force on the test specimen, the impact energy used was the most that could be obtained using plant air at 120 psi (827 kPa), just under 60 Joules.

The panels were heated using the radiant burner and a propane/air mixture. The burn time was approximately 15 minutes. Temperatures of 600° - 700°C were used for the thin panels, and between 800° and 900°C for the thick panels. The temperatures were measured at the

hottest point on the sample surface. During the burn period a flow of air was maintained in the test chamber through a filter to collect any light debris that was emitted. The resin was typically consumed in the burn area in a matter of seconds. At the end of the burn period a new filter was placed in the air-stream and the specimen impacted. As a result of this impact, a few hundred milligrams of material were typically found on the bottom of the test chamber. Only the lightest particles were deposited on the air filter during the fifteen minute period under continued air flow following the impact of the specimen. These often included very long single graphite fibers for the less effective and all graphite control configurations. The weight of fibers collected on the filter was measured and used to rank the panels tested.

For the screening study three considerations were used to evaluate the relative merit of the candidate configuration tested. These are as follows:

- a) The quantity of graphite fibers collected on the filter during and after the impact test following burning.
- b) The visual appearance of the panel after impact.
- c) The nature and quantity of the debris collected from the bottom of the test chamber after impact.

For the glass/graphite hybrid constructions, a quantity of clean glass fiber was also collected on the air filter as was a varying quantity of soot particles. The graphite fibers collected on the

filter were often the smallest fraction of the total material and ranged in length from approximately one to 10 millimeters. For screening purposes, if the quantity of graphite fibers collected exceeded five to seven milligrams, the panel configuration was rejected. Unprotected graphite panels typically deposited more than 20 milligrams of graphite fiber on the filter.

It was not unusual to find several grams of material on the bottom of the test chamber. This material might include panel fragments and bundles of fiber in various stages of decomposition. This material was ejected from the panel on impact, but was too heavy to be transported by the airstream to the filter. The weight of this ejected debris was determined as well as the type and nature of the materials present.

One early result of the burn/impact screening study was that the size of the specimen and the orientation of the principal graphite reinforcement could greatly affect the outcome of the test. To exemplify these conditions, three specimens of a panel of the configuration A-3B-A, IDCC-11, are shown following burn/impact testing (Figures 6 through 8). In Figure 8 the unidirectional fibers are transverse to the specimen length (specimen measured 25 by 102 mm [1X4 inches]). The fibers found on the air filter can be seen on the left side of the photograph, while those from the bottom of the tester are on the right. A great quantity

of the 25mm long fibers could be seen floating in the air after the impact. The results of testing the same panel configuration is in which the graphite fibers were parallel to the longitudinal direction of the specimen are shown in Figure 9. Again the specimen was a 25 by 102 mm coupon tested in the same way. The fibers collected on the air filter are shown in Figure 9, and weighed only a few milligrams. Also shown in this figure is the material collected from the bottom of the tester, very different in nature from that of the transverse fiber construction of Figure 6. When the width of the specimen was tripled, there were essentially no fibers released in the test. The tested specimen of this size is shown in Figure 8.

That even a single ply of glass cloth afforded some protection is apparent by comparing the results of unprotected panels, such as the one shown in Figure 10, with those just discussed. The use of the glass cloth lowered the quantity of graphite fiber released in the burn/impact test. As shown in Figure 11, the Kynol cloth (phenol-formaldehyde) did not have the same effect. The Kynol burned away from the graphite in the central area, leaving the graphite fiber totally unprotected.

Panels utilizing novolac epoxy resins were found to burn more vigorously than those based on tetraglycidyl methylene dianiline (TGMDA). This increase in flammability led to more destruction of the panel in the burn/impact test, and hence to a

greater release of fiber than that from a comparable TGMDA based epoxy panel. The replacement of the epoxy with the polyimide PMR15 led to significantly better results. A sixteen ply uni-directional graphite panel was fabricated with PMR15 and tested with the fibers in the transverse (short) direction of a 25 by 102 mm specimen. The results of this test are shown in Figure 12. In the impact portion of the test just two pieces fell out which contained the single fibers quite solidly. The air filter was found to contain only one milligram of carbon particles. In this way a significant improvement was obtained through the use of a more thermally capable resin system.

Approximately thirty candidate hybrid panels were fabricated for screening, from which twenty were selected for further study. The configuration and calculated thicknesses of these are shown in Table 4 for the thin panels, and Table 5 for the thick panels. The results of burn/impact testing of these configurations are presented in Section 2.2.2.

2.1.4 ADDITIVE SCREENING STUDY

One of the areas investigated in this program was the use of additives within the composite structure to increase the ability of the laminate to retain its graphite fiber primary reinforcement. A few preliminary panels were fabricated for the burn/impact screening test described above in which non-structural

layers were incorporated within the composite. The use of Kynol phenol-formaldehyde polymer cloth (described above) did not afford any protection of the graphite. The char did not bind the fibers together to prevent their release.

Another trial was done with layers of phenolic microballoons (BJO-0930) used between layers of unidirectional graphite/epoxy. Again, no adhesion of the char was observed with the graphite, and no protection of the panel was obtained. A trial with glass flakes yielded similar results. On burning, the glass flakes did not adhere to the graphite fiber, so that the protective capabilities of glass were not realized in the structure. Glass cloth was found to offer much better protection as it had structural integrity on its own after the resin had burned away.

Another approach to the use of additive materials was to incorporate them into the resin itself. To expedite the testing of a large number of potential chemical additives for the resin, a simple experiment was devised to screen these materials and determine their effect on the graphite fiber. The procedure used was to start with one inch square single plies of graphite cloth placed on a ceramic plate. A suspension was made of the finely ground compound in epoxy resin. This suspension was coated onto the ply so that approximately three-fourths of the ply was covered with a progressively thicker layer of the material.

The uncovered area was used as a standard during the heating of the material. The specimens were heated from ambient to approximately 1200°C in an electric muffle furnace. Each sample was observed periodically throughout a total span time of two to three hours.

In this fashion more than forty different compounds were screened. Two of these compounds were found to greatly alter the burning behavior of the graphite. These two were magnesium oxalate, and the mineral ulexite.¹ The magnesium oxalate was found to accelerate the burning rate of the graphite. Even the use of relatively small quantities appeared to cause the graphite to disappear at lower temperatures. Most of the work on the burning of composite panels was done in the 600° to 900°C range, and at these temperatures the effect of the magnesium oxalate on assisting the fiber to burn away was small. As a result, the study of composites with this salt was dropped.

The effect of the ulexite was significantly more important. Ulexite

1 A patent is pending for the use of ulexite to reduce flammability and improve the containment of materials.

prevented the fibers from burning at any temperature tried, including up to six hours at 1200°C. The material even migrated to cover the uncovered portion of the graphite cloth. Observation of the fibers after heating showed that they were encapsulated in a glassy material that effectively bonded the fibers together and prevented their burning. The glass formed was quite brittle, but kept the graphite in clumps, rather than as single fibers, when broken up.

Other compounds were found to modify the burning behavior of the compositing materials, but none to the same degree as the ulexite. The following materials were found to retard burning: boric acid and its anhydride, colemanite, meyerhofferite, apophyllite, ulexite, sodium sulfide, and Firebrake 2B (a synthetic zinc borate). A slight enhancement of the burning rate of the graphite was observed after adding copper oxalate, aluminum chloride, zinc acetate, manganese dioxide, and potassium permagnate. The temperature at which the ulexite began to form its protective glass coating was much higher than the burn temperature of the epoxy resin. The addition of boric acid or boric anhydride to the ulexite reduces the temperature of glass formation allowing the ulexite to be very effective in actual composite panel use.

Table 6 contains comparative data for burn/impact testing of some configurations with and without the ulexite/boric acid additive. For a configuration that is already protected by layers of glass

cloth, the additive has little effect on the weight of material collected on the air filter. In all cases, however, the graphite fiber in the panels containing ulexite tends to stay together. For unprotected panels the results are very dramatic. For the all graphite Ekl configuration a ten fold decrease in the quantity of graphite collected on the air filter is observed. This changes an unacceptable configuration to an acceptable one with no secondary reinforcement used. Only preliminary comparisons were made of the strength of panel configurations with and without this additive system, but these showed promise of a minimal penalty to mechanical properties as long as the particle size of the additive materials was kept small, as shown in Table 7. Further work beyond this program would be needed to more fully characterize the effect of this additive system on the overall performance of a composite structure.

2.1.5 LAMINATE ANALYTICAL STUDY

As part of the laminate screening of Phase I of the program, CH used the computer program "LAMSTIF1" to establish the stiffness characteristics of candidate configurations of materials. This program could handle the calculation of these properties for a wide variety of hybrid materials and structural configurations. Factors such as grouping of the plies within a given configuration to allow for preplying operations in manufacture were favored wherever possible to maximize the ease of fabrication.

Structures with the greatest interspersing of different materials would be more costly to control and use in a production environment. The selection of glass as the secondary reinforcement of choice was based on its oxidative stability, high strength, and low cost. Various forms of graphite fiber, as tape, cloth, and hybrid cloth, were considered throughout the program. Tables 8 and 9, for the thin and thick panels, respectively, summarize the longitudinal, transverse, and shear moduli, and Poisson's ratios calculated for eighteen of the configurations considered. Except for the cases of primarily forty-five degree reinforcement and balanced 0/90 reinforcement, all of these configurations have longitudinal tensile modulus values in excess of the ten million psi criterion.

2.2 FABRICATION AND EVALUATION OF SELECTED CONCEPTS

In this phase of the program, laminates of the selected concepts were fabricated and tested for burn/impact performance, as well as for mechanical and physical properties. At the conclusion of this task the laminates for delivery to NASA were fabricated.

2.2.1 LAMINATE FABRICATION

The laminates fabricated in this phase of the program were all prepared by autoclave processing. All of the unidirectional graphite/epoxy tape used was in the form of prepreg, including prepreg

based on Narmco 5208, Hexcel F263, or Fiberite 934 resin. The 7781 style glass cloth used was typically in the form of prepreg as well, in Narmco 5208 or Ferro CE9000 resin. All of the graphite cloth and the 120 style glass cloth used were impregnated by CH. The coating was accomplished using solutions of CH4010, F263, or 934 resin. The 104 style glass was used dry in the panel layup and became impregnated during the cure cycle. Additives to the resin were incorporated by mixing them into the solvent solutions used to prepare the prepreg. The resin content of all of the internally made prepreg was controlled by weighing the reinforcement and impregnating with a calculated weight of resin. The excess solvent was removed, to below 2% by weight, by air drying and low temperature (150°F typically) oven drying, prior to the use of these materials in the panels. The materials selected for a given panel were cocured in 350°F, 100 psi autoclave cycle.

In the screening work the Kevlar cloth was used as a prepreg in 5208 resin. The Kynol, phenol-formaldehyde cloth was impregnated at CH. When it was necessary to cocure resins of very different viscosities, some preconditioning (staging) was done on the more flowable of the group so as to control the resin flow during final cure. This staging was usually done at 250°F for period up to one hour in duration. All of the panels were molded on a stainless steel caul in a two foot by four foot autoclave. Up to ten panels

were molded at one time under the same vacuum bag, and conventional materials were used in the layup for cure.

All of the operations involved in the fabrication of the test panels were controlled by detailed instructions provided to the shop technicians actually doing the work. In this way the fabrication of these panels was made to simulate the procedures that would be used in the fabrication of a piece of actual hardware.

2.2.2 FLAMMABILITY/IMPACT TESTING

Following the screening studies discussed above, laminates were fabricated and tested in the burn/impact tester to determine their tendency release fibers. The results of these tests on the selected concepts, and some comparative results on panels from the screening study are provided in Table 10 for the thin panels, and in Table 11 for the thick panels. The notes appended to these tables describe some of the differences in testing and results among the configurations listed.

The use of glass cloth as a surface barrier was found effective in the thin panels in reducing the release of graphite fibers (see Figures 10 versus 7). For the thick panels a heavier layer of glass cloth at the surface afforded even better protection. In Figure 13 a considerable degree of destruction can be seen due to impact of configuration is DL-23, yet the air filter contained only five milligrams of fiber, as shown in Figure 14.

Less panel destruction is apparent in the photographs of the test performed on configuration CD-5, (Figures 15 and 16). The fibers collected on the air filter again weighed only five milligrams. The fibers, mainly glass, and other ejected debris from CD-5 collected from the bottom of the tester are shown in Figure 17. As discussed in the notes with the tables (10 and 11) several temperature conditions were tested, as well as differences in specimen configuration. In general it appeared that more intimate mixtures of glass cloth and graphite were more successful, especially in the thin panels. Differences between interlaminar mixing of glass and graphite and intralaminar mixing could not be readily distinguished in the testing done in this program. Figures 20 50 33 present photographs of the balance of the various ply configuration panels tested for resistance to fiber release.

2.2.3 MECHANICAL/PHYSICAL PROPERTY EVALUATION

All of the panels fabricated were subjected to tests to determine their physical and mechanical properties. The physical testing included acid digestion for resin content, fiber volume, and void content, ultrasonic examination, and metallographic examination. The acid digestions were done in hot sulfuric acid with subsequent addition of hydrogen peroxide. The basic method employed is based on ASTM D3171, including specific gravity determination in distilled water with a drop of surfactant, Zephiran Chloride.

The ultrasonic examination was done by C scan techniques at 5 MHz. For the thin panels 26dB was used and 34dB for the thick panels. Calibration was accomplished in all cases by means of 0.25mm (0.01 in.) thick lead foil tape as a standard. Figure 32 displays the results for four panels showing no indications of defects, except for the lead tape standards. This result was entirely typical of all of the panels fabricated for Phase II of the program.

Metallographic examination was performed on all of the panels as well. Sections were cut using an abrasive cutoff saw and polished for microscopic examination. A Leitz Wetzlar Metallographic Microscope MM5 was used for this purpose. Photographs were taken of the polished sections using Type 52 polaroid film. Figures 33 through 39 display sections taken from thin panels of the study, and Figures 40 through 45 sections of thick panels. The degree of inter and intralaminar mixing of the primary and secondary reinforcements is clearly apparent in these micrographs. They also show the fabricated panels to be typically void-free, bearing out the results obtained by ultrasonic examination.

Short beam shear (SBS) and flexural testing were performed on the panels. The shear testing was done in accordance with ASTM D2344, except that the support rod diameters were the same as the loading nose diameter in 6.35 mm (0.250 in.). All of the shear tests were done at a span to depth ratio of 4:1. The flexural tests were done in accordance with ASTM D790 with flexural modulus being determined

using crosshead motion corrected for machine deflection under the same loads and temperatures as used in the tests. Both types of testing were performed at ambient temperature (RT) and at 450 K (350 .F) using an Instron Universal test machine. The results of these tests are provided in Tables 13 through 15.

2.2.4 FINAL CONCEPT SELECTION

Based on the results obtained in Phase II, panel configurations were selected for delivery to NASA in the form of eight inch by eight inch panels. The emphasis in this selection was on configurations that performed well in the burn/impact tests and were relatively simple to fabricate. The selected configurations are presented in Table 16. A panel representative of each of these configurations was fabricated using autoclave processing as described above, and delivered to NASA Lewis Research Center. The panels were examined by ultrasonic examination and none of the panels were found to have ultrasonic indications of any defects.

3.0 CONCLUSIONS

While there are conditions of combined fire and impact that could lead to the release of single graphite fibers, modifications to the structure of the composite and its constituent materials can be made to reduce this effect. Glass/graphite hybrids offer a simple approach that greatly reduces the quantity of fibers

released. More thermally capable resins like the polyimide, PMR15, appear to offer more direct advantages in the protection of the composite without the use of a secondary reinforcement. Certain additives were also identified that could be incorporated into epoxy resin based composites that nearly eliminated single graphite fiber release by agglomerating the fibers in a high melting glass-like material. While studies show the risk of damage from graphite fiber release to be small, the hybridizing of the composite structure has been found to lower the tendency for the fibers to be released.

REFERENCES

1. "A Report of Observed Effects on Electrical Systems of Airborne Carbon/Graphite Fibers", NASA Technical Memorandum 78652, Jan. 1978.
2. Carbon Fiber Risk Analysis, NASA Conference Publication 2074, Oct 31-Nov 1, 1978.
3. Published at Technology Conference on Advanced Composites - Special Topics (Dec 4-6, 1979 El Segundo, CA) by Bill Gilwee and Richard Fish.

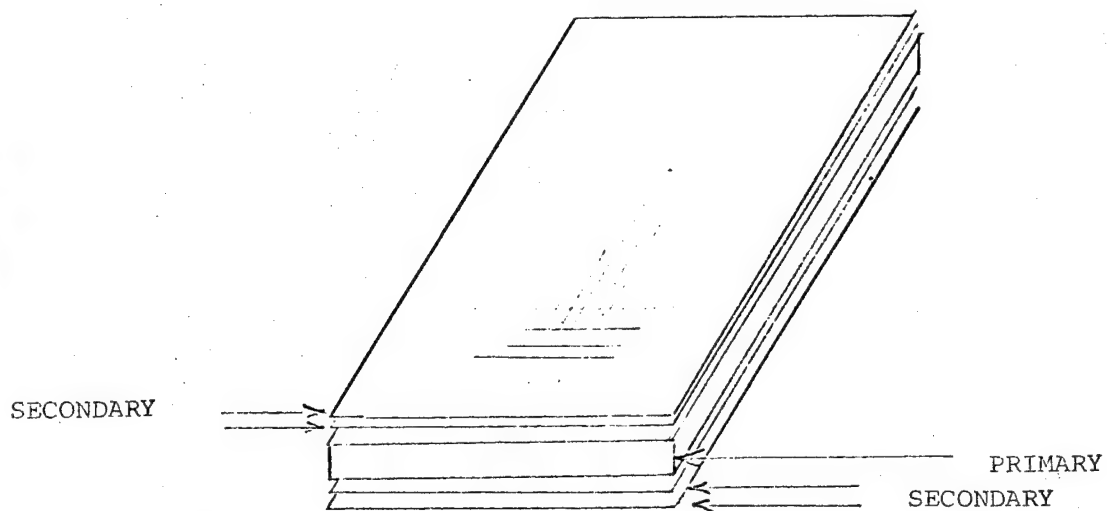


FIGURE 1. HYBRIDIZING CONCEPT, SURFACE PLIES

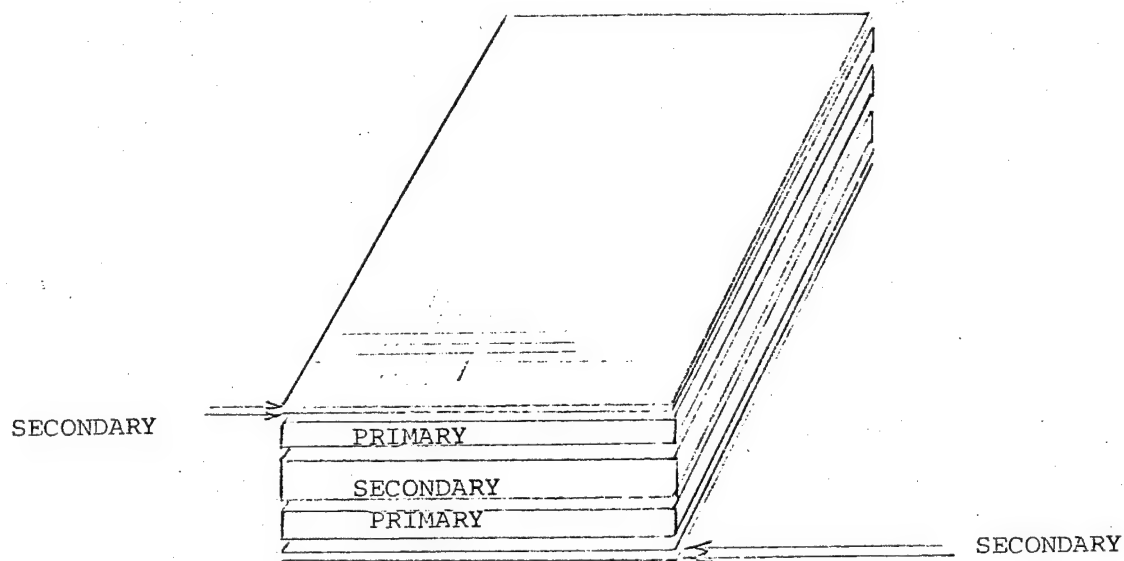


FIGURE 2. HYBRIDIZING CONCEPT. CENTRAL CORE AND SURFACE PLIES

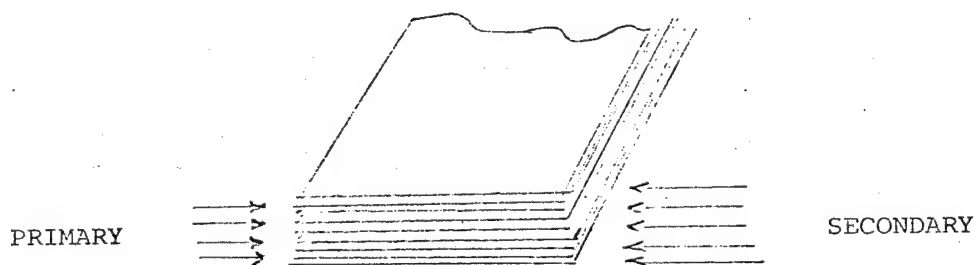


FIGURE 3. HYBRIDIZING CONCEPT. FULL INTERLAMINAR MIXING

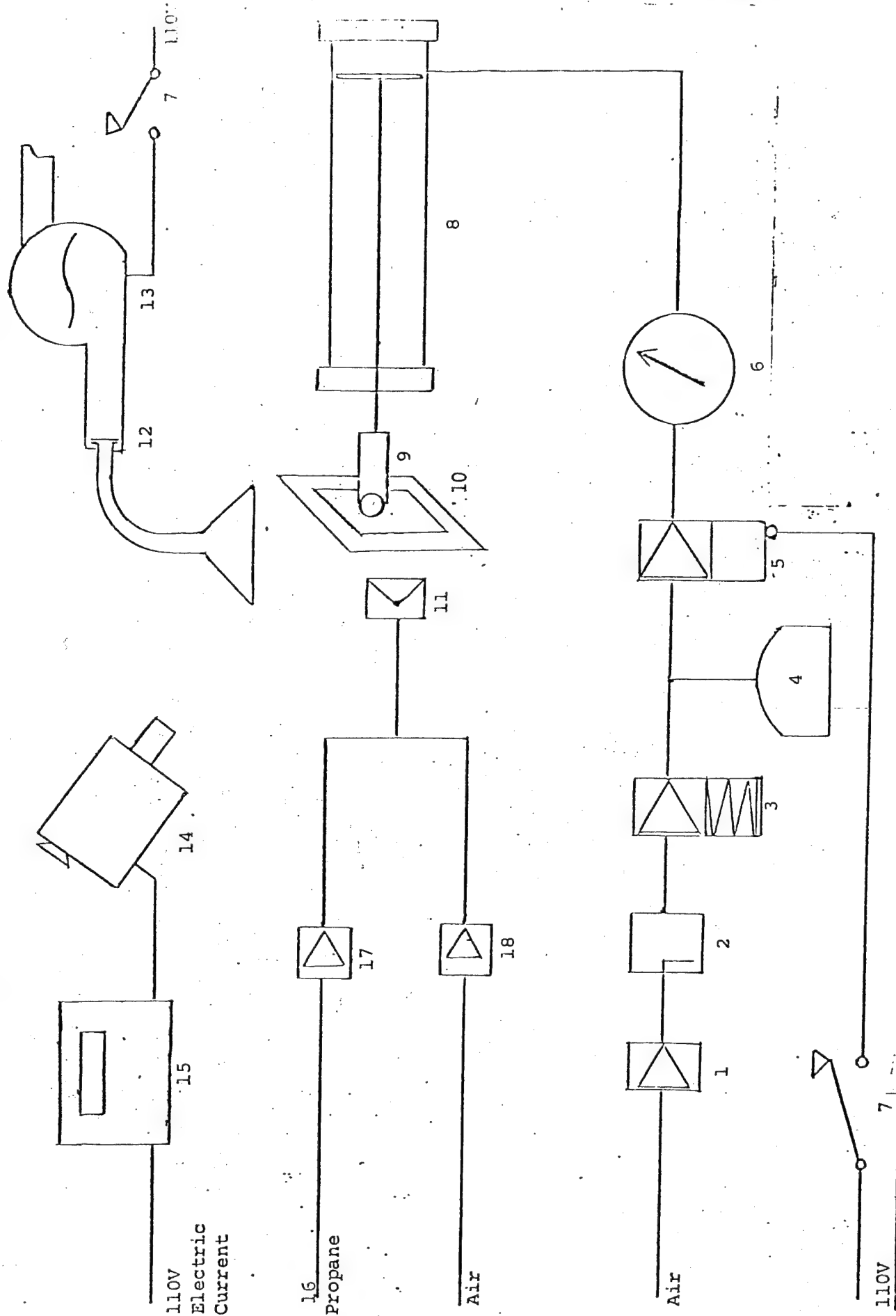


FIGURE 4 . SCHEMATIC OF BURN-IMPACT TESTER

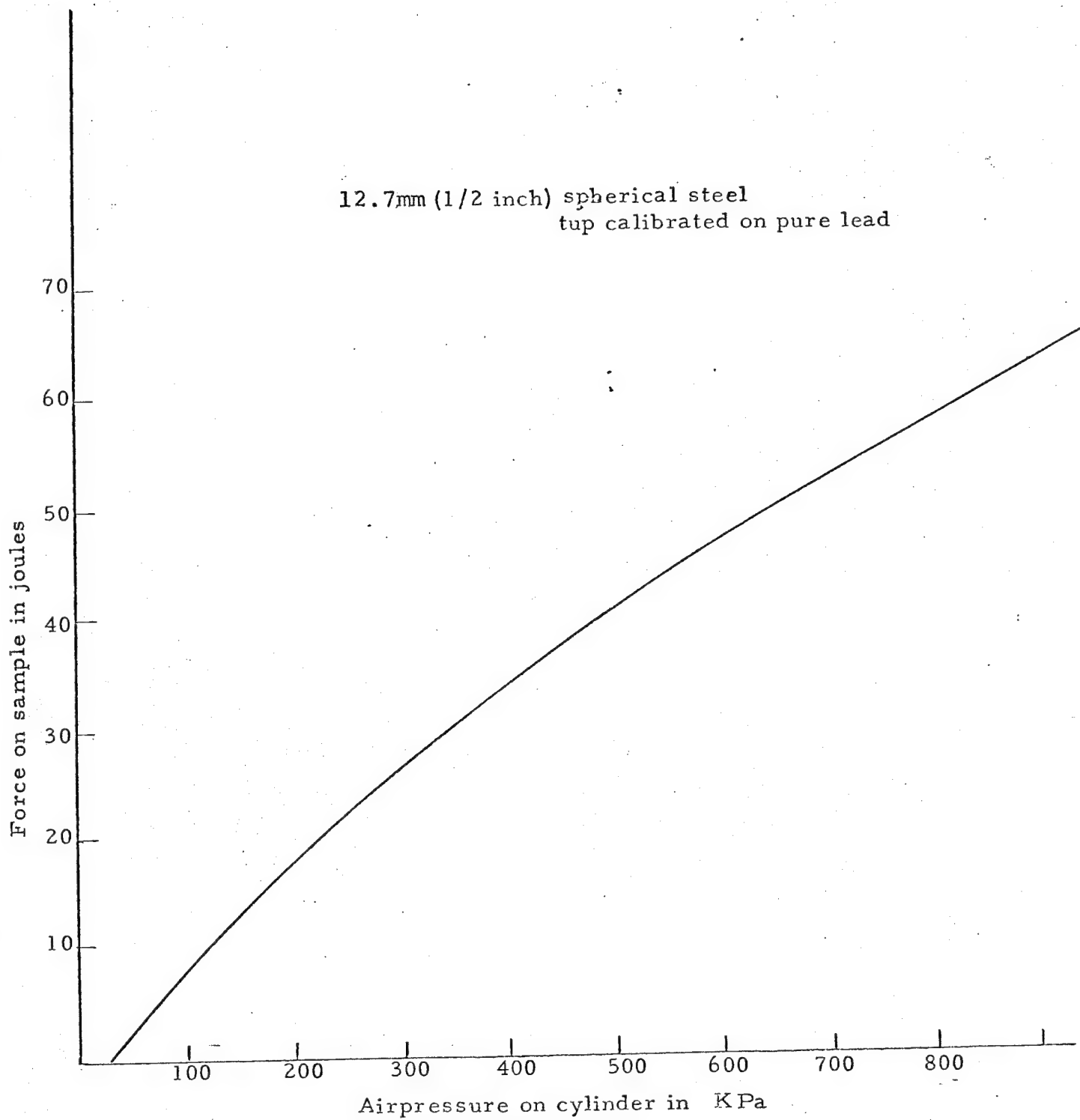


FIGURE 5 . CALIBRATION CURVE OF IMPACT TESTER

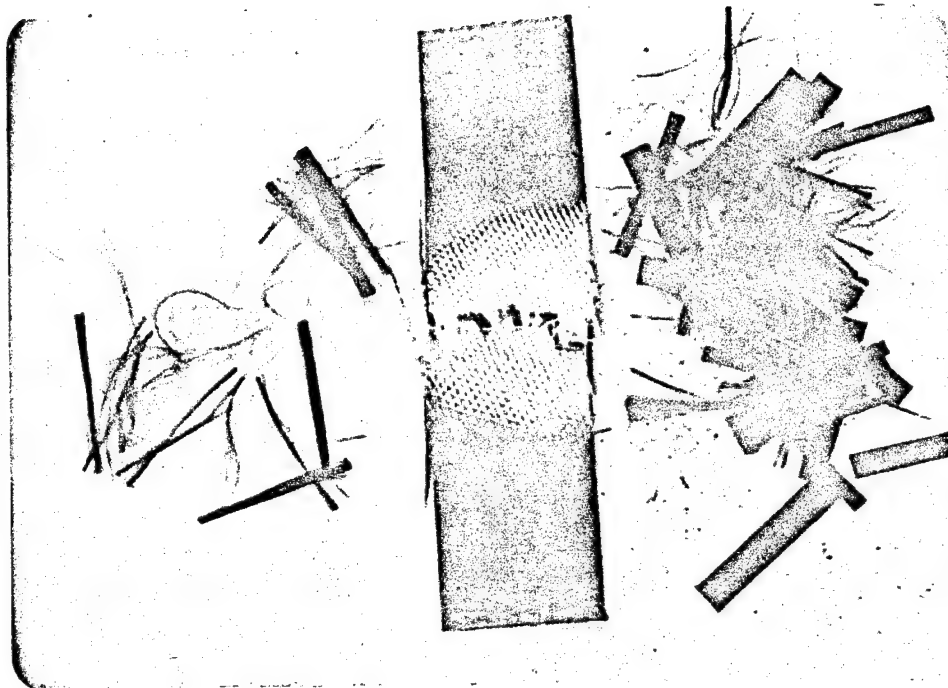


FIGURE 6. SAMPLE FROM PANEL CC-11 FRONTVIEW
AND FIBERS RELEASED BY BURN/IMPACT
TESTING. (TRANSVERSE GRAPHITE
FIBERS)

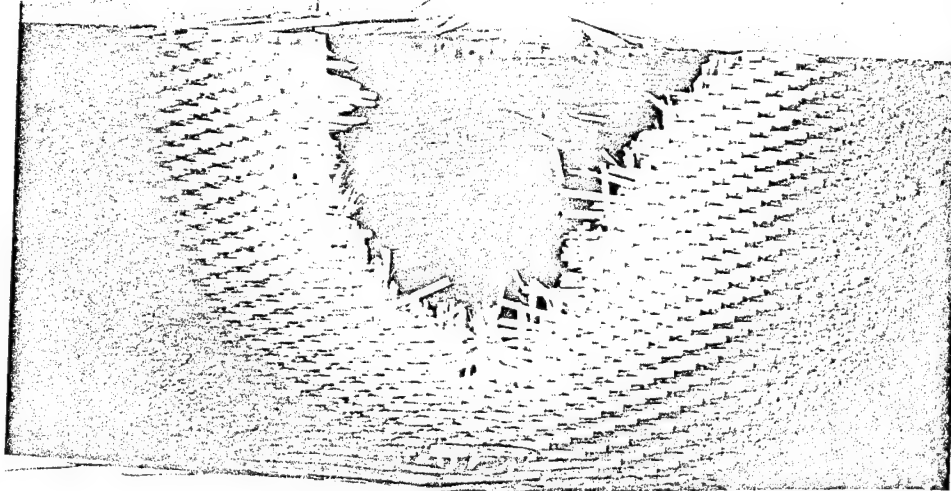
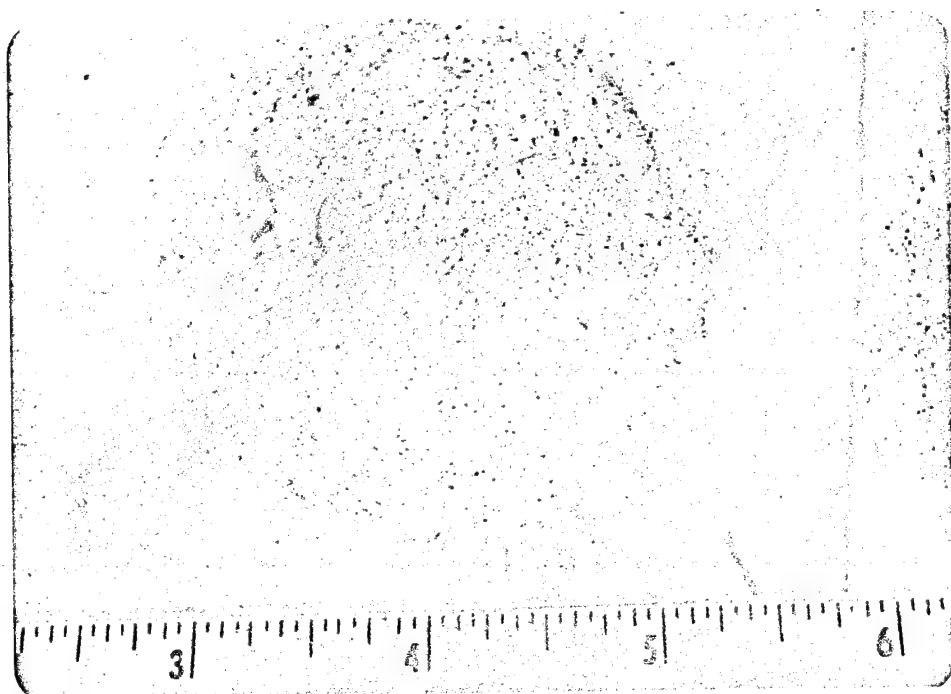


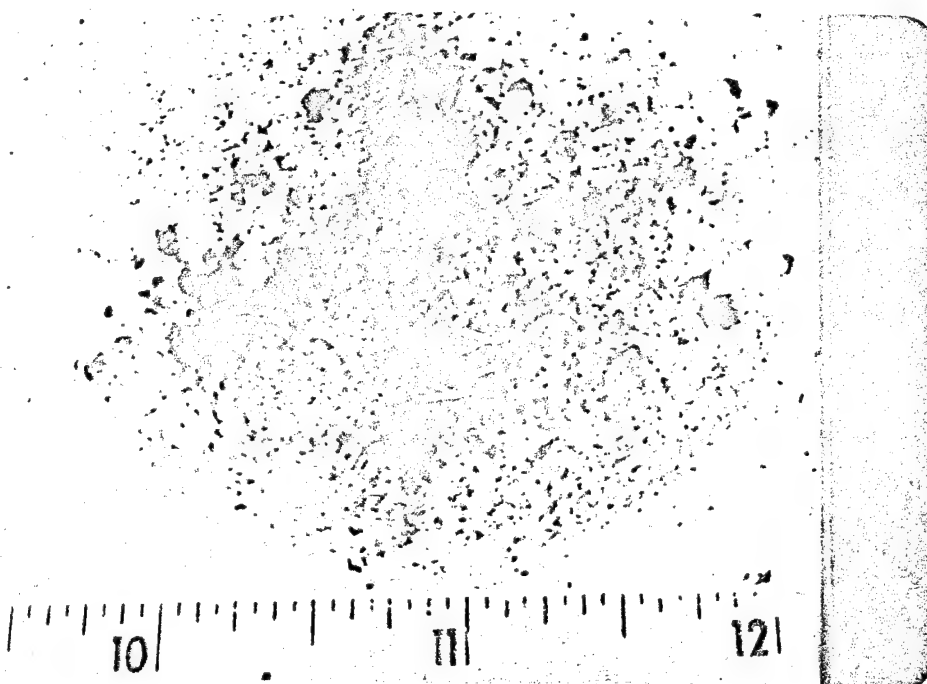
FIGURE 7. FRONTVIEW OF SAMPLE OF PANEL CC-11
(LONGITUDINAL GRAPHITE FIBERS)



FIGURE 8. REARVIEW OF SAMPLE OF PANEL CC-11
(3 X 4 inch)



Fibers on air filter



Fibers on bottom of tester

FIGURE 9. COLLECTED EJECTA FROM TEST OF CC11 SAMPLE FROM FIGURE 9.

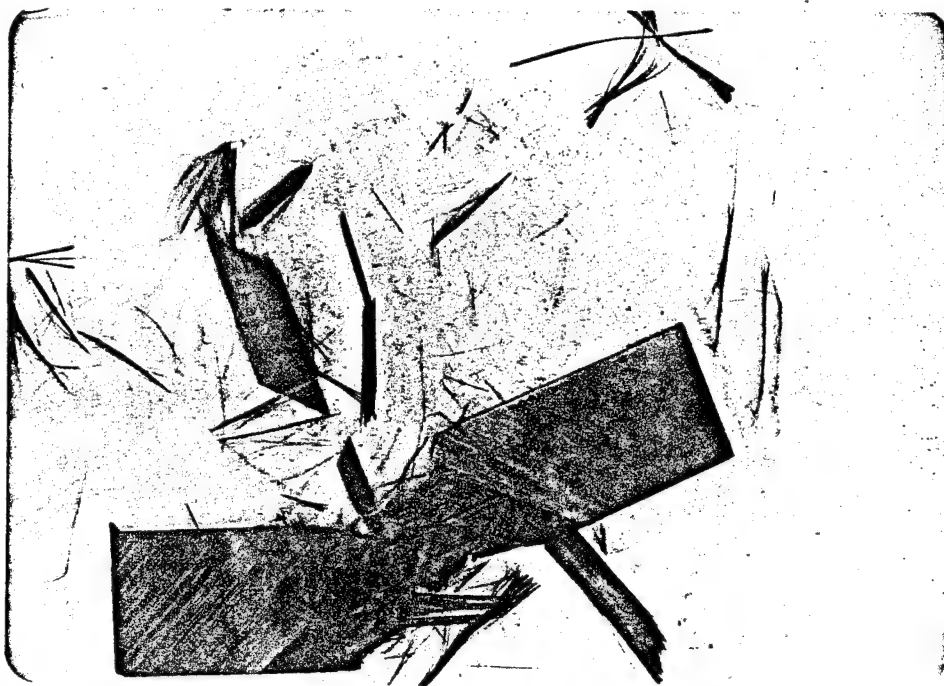


FIGURE 10. TEST RESULTS OF UNPROTECTED GRAPHITE/EPOXY PANEL EK1 SAMPLE

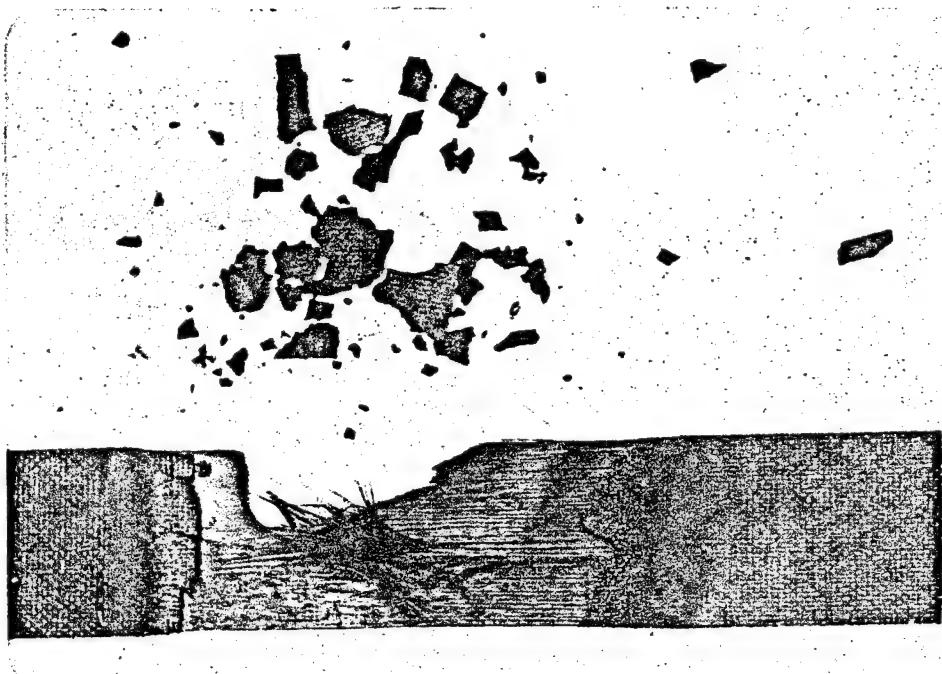


FIGURE 11. SAMPLE AND EJECTA OF PANEL EK2 (KYNOL FACED PANEL)

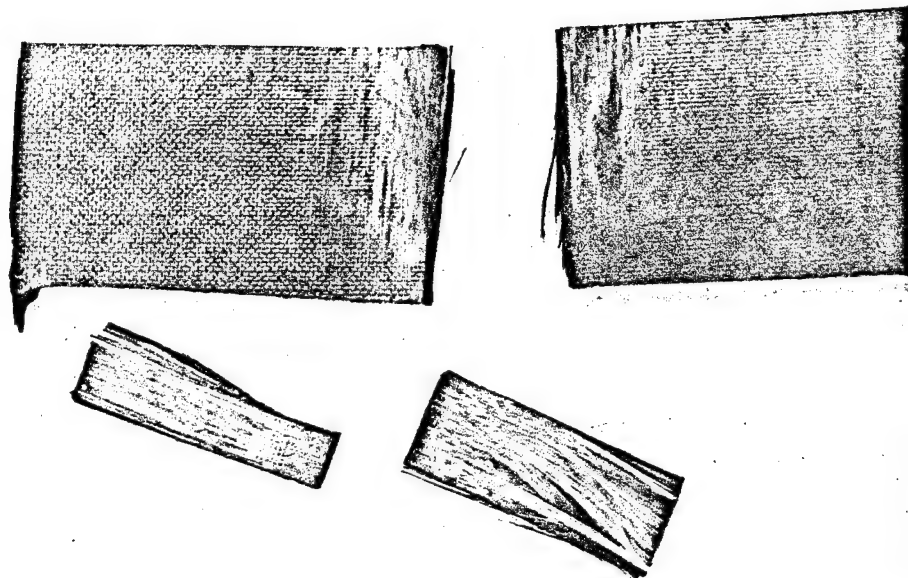


FIGURE 12. TEST RESULT OF GRAPHITE/
PMR15 COMPOSITE

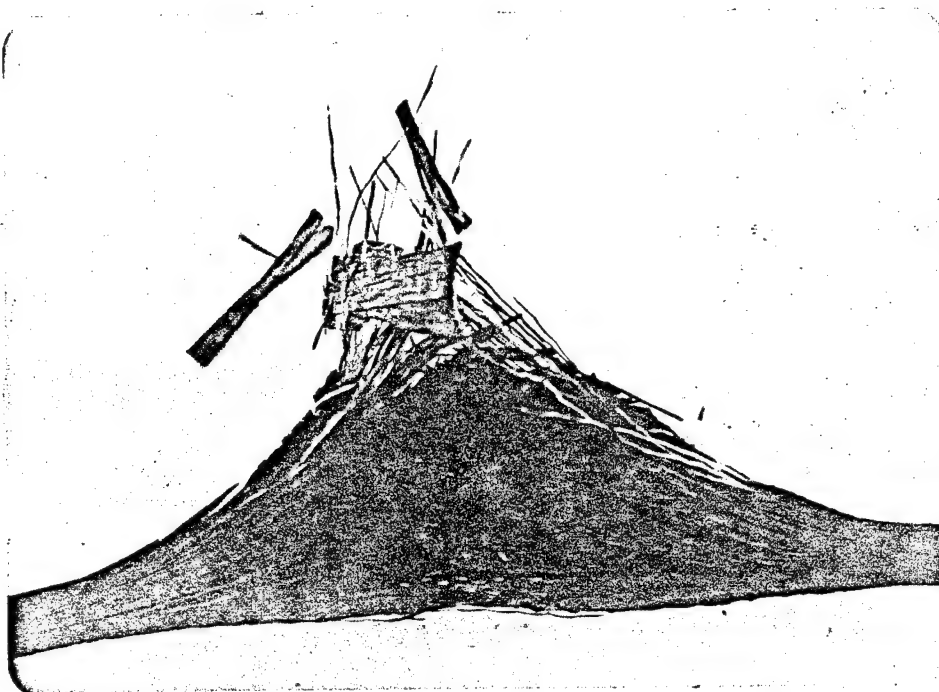


FIGURE 13. EFFECT OF IMPACT ON BURNED
PANEL DL 23

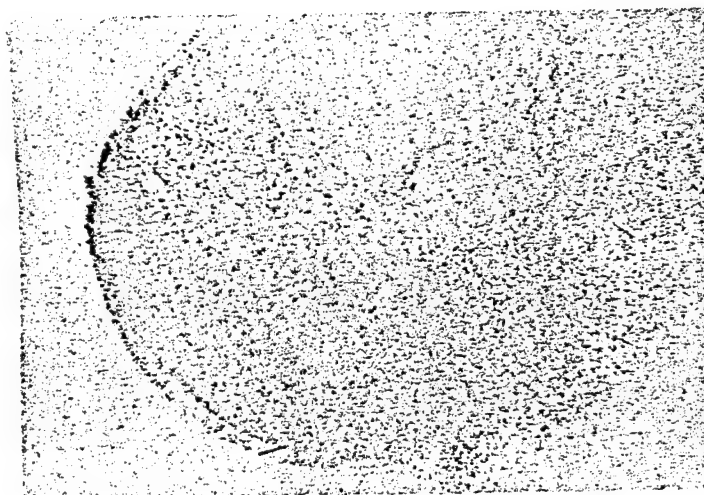


FIGURE 14. CARBON PARTICLES ON AIRFILTER,
SAMPLE FROM DL 23 (See Figure 15)

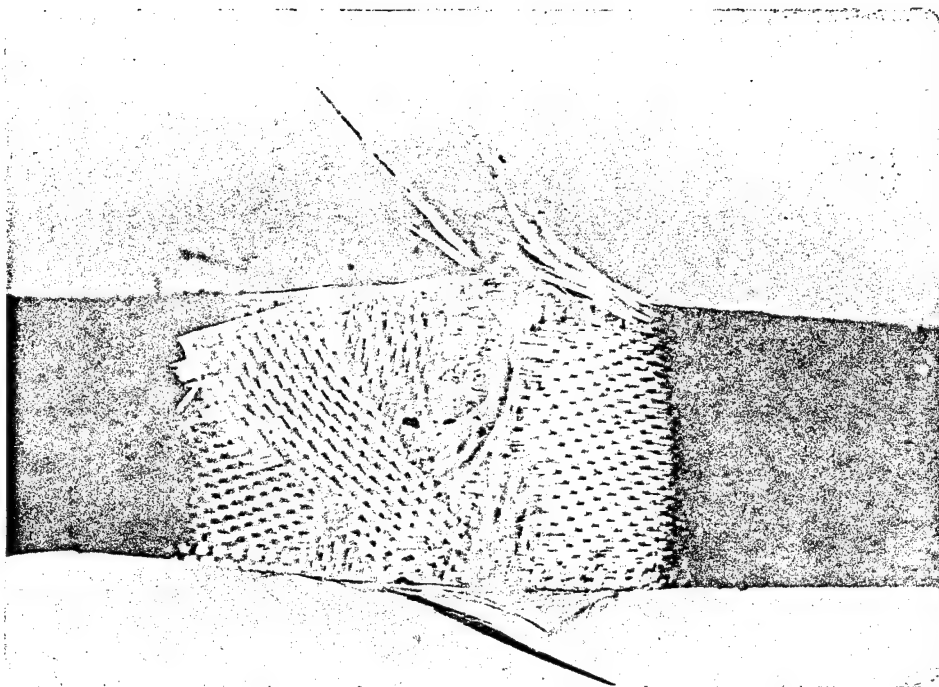


FIGURE 15. FRONTVIEW OF SAMPLE OF CD-5

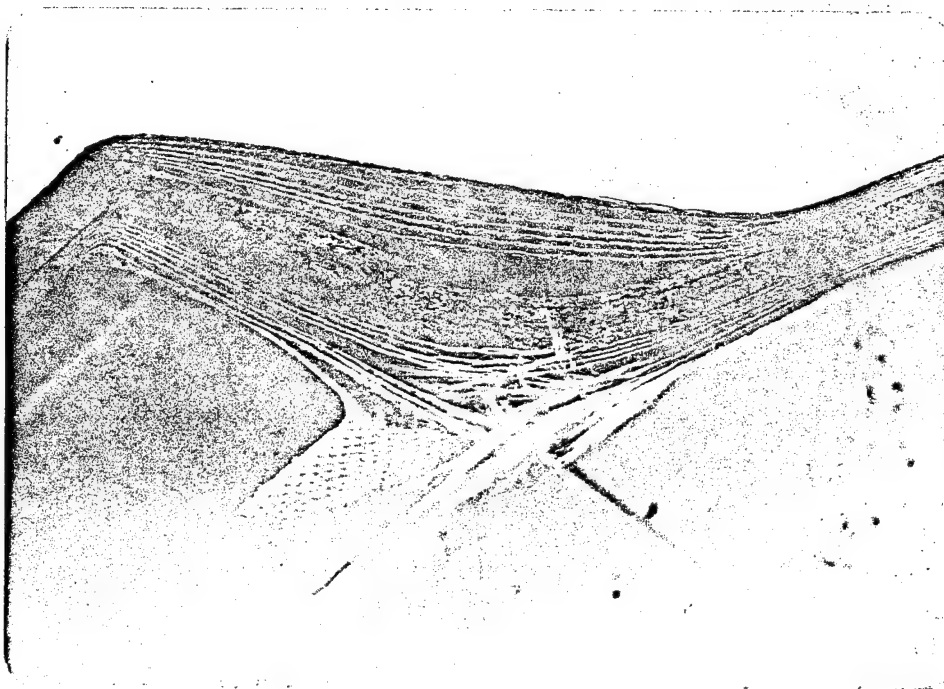


FIGURE 16. SIDEVIEW OF SAMPLE OF CD-5

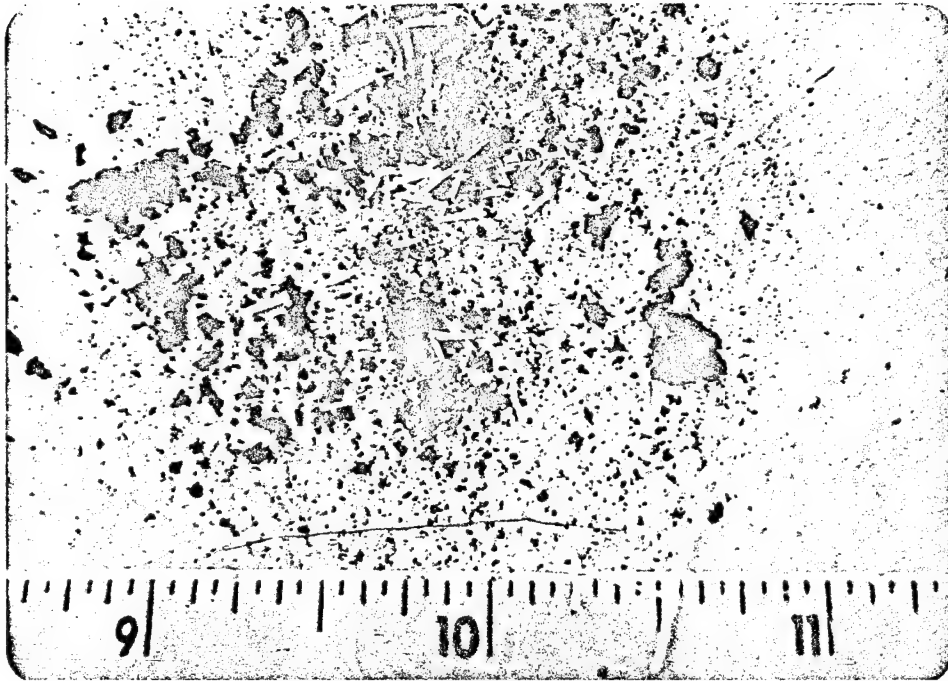
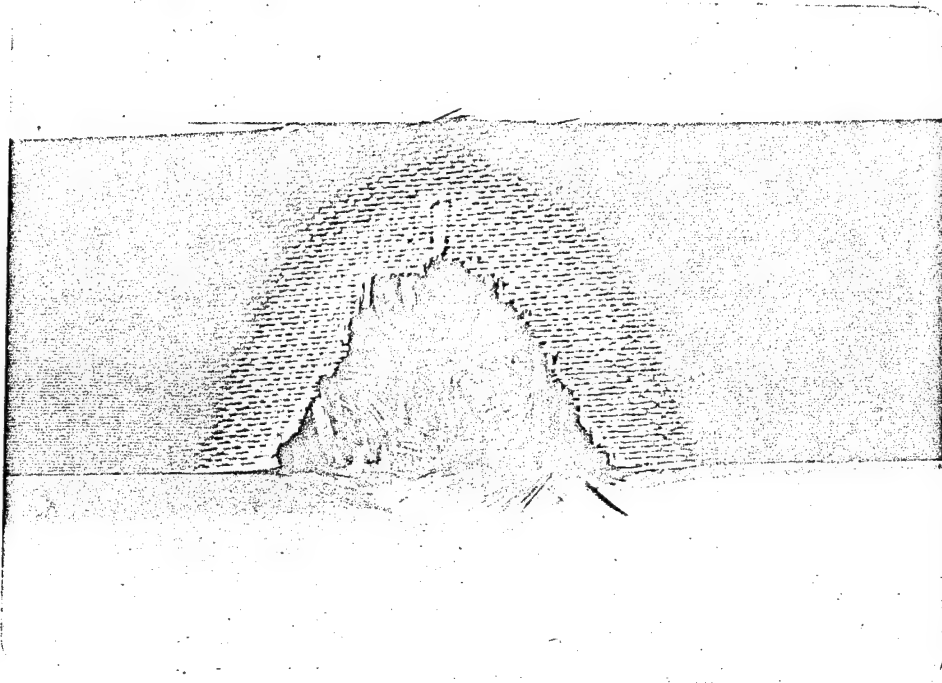
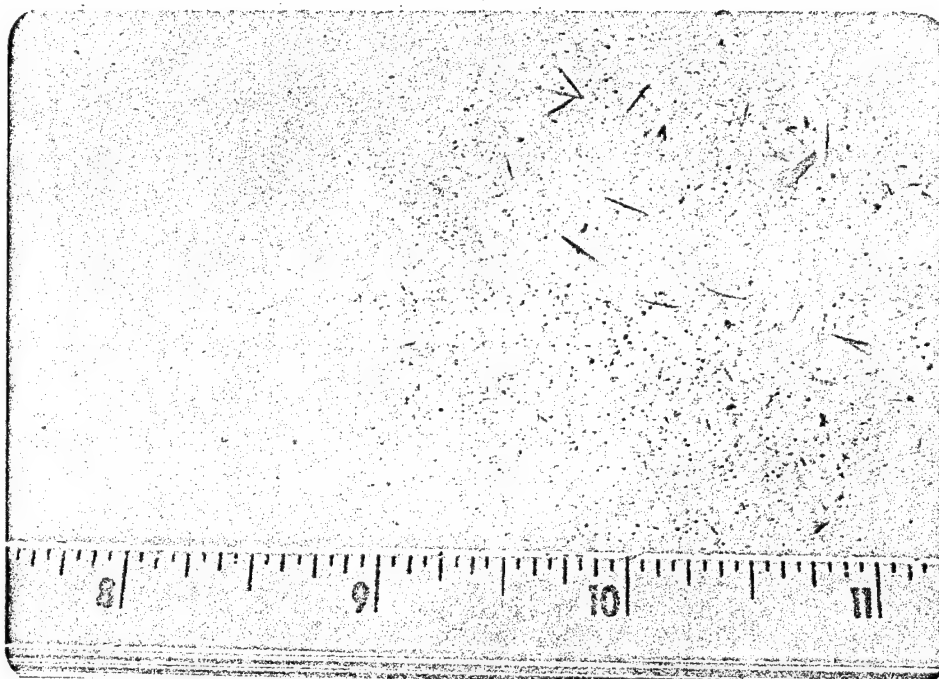


FIGURE 17. EJECTA FROM BOTTOM OF TESTER
CD5 SAMPLE



Frontview and



fibers from airfilter. Sample comes from CH-8.

FIGURE 18. TEST RESULTS OF CH-8.

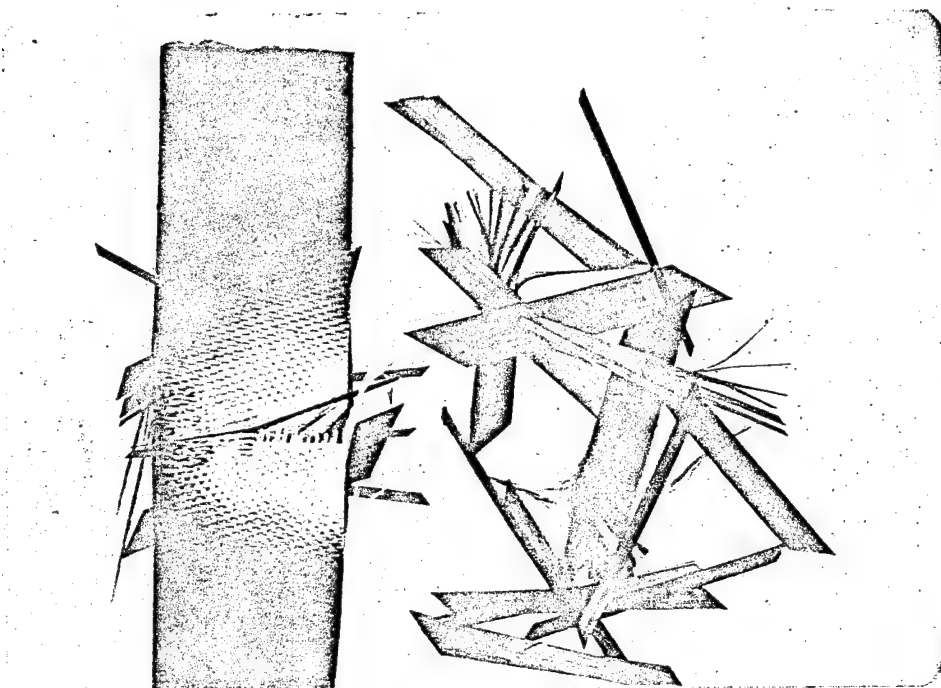
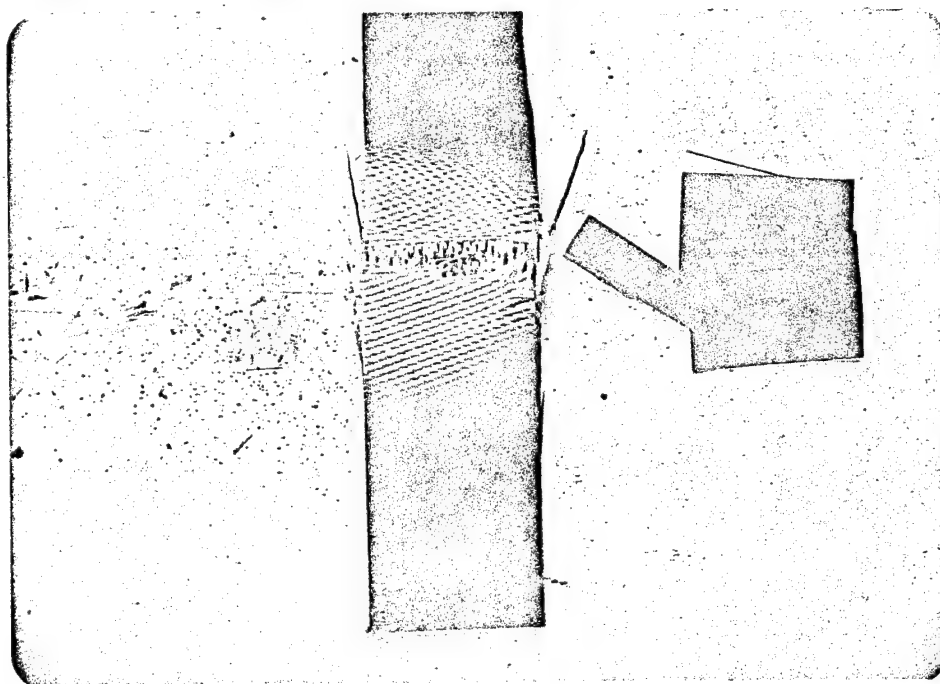


FIGURE 19. SAMPLE OF CONFIGURATION DL-18.



From left to right: airfilter residue, frontview of sample, and pieces from panel CC-34.

FIGURE 20. TEST OF CC-34.

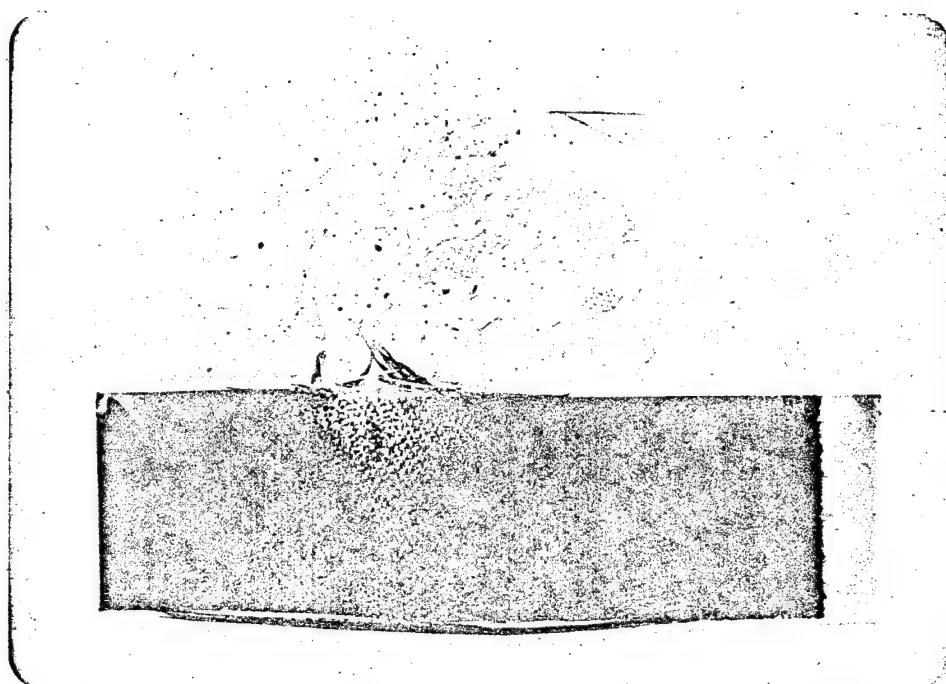
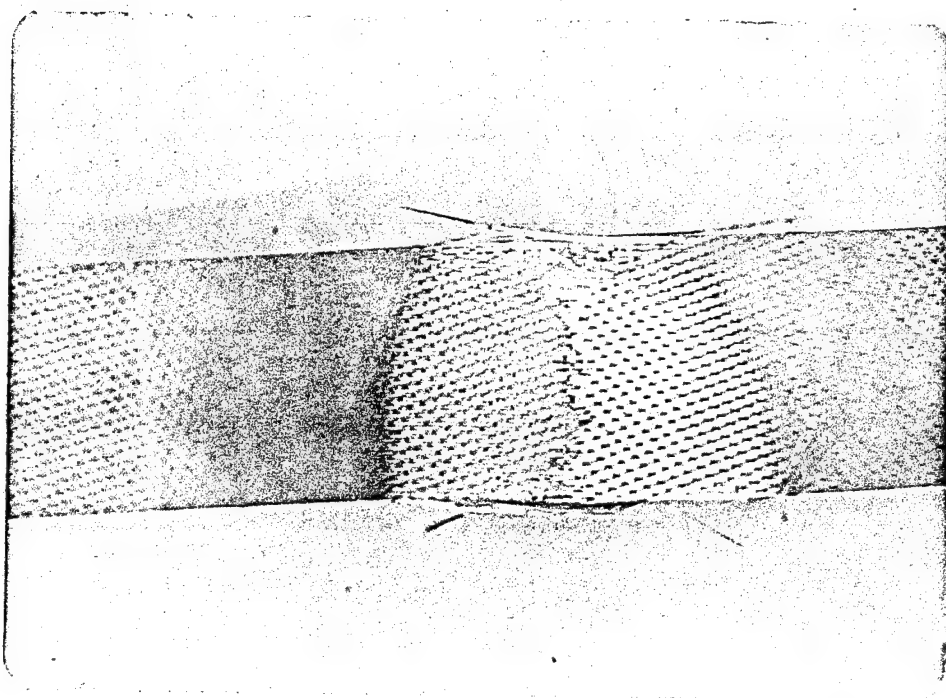
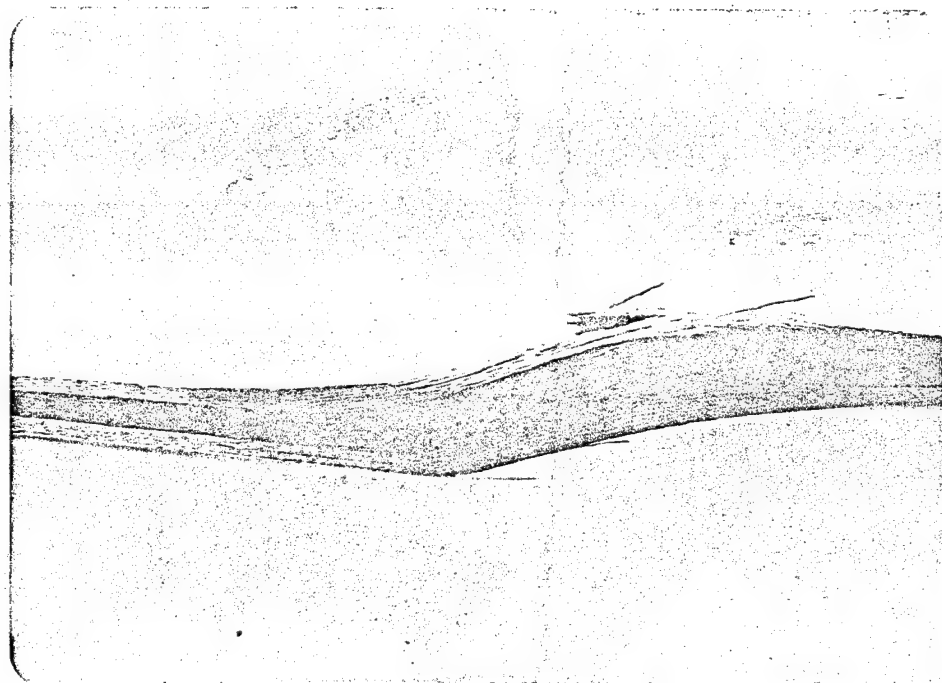
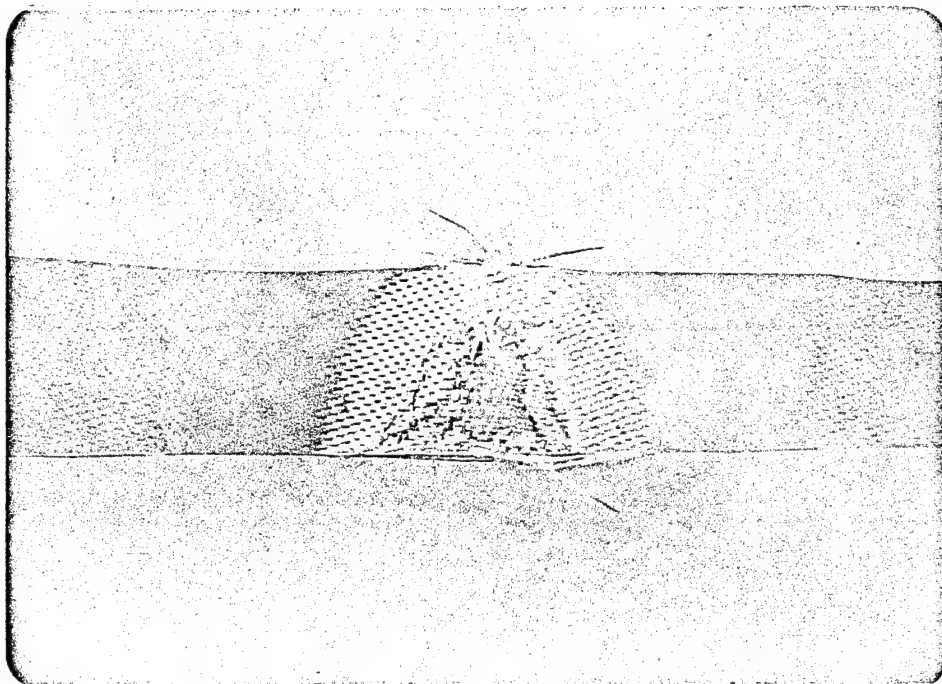


FIGURE 21. SAMPLE FROM DL-27. REARVIEW AND RELEASED FIBERS AFTER BURN AND IMPACT TESTING.



Burn and impact testing frontview of panel and fibers
from airfilter, panel CH-10.

FIGURE 22. TEST RESULTS CH-10.



Front and side view of sample from panel CH-5 after burn-impact testing.

FIGURE 23. SAMPLE FROM CH-5

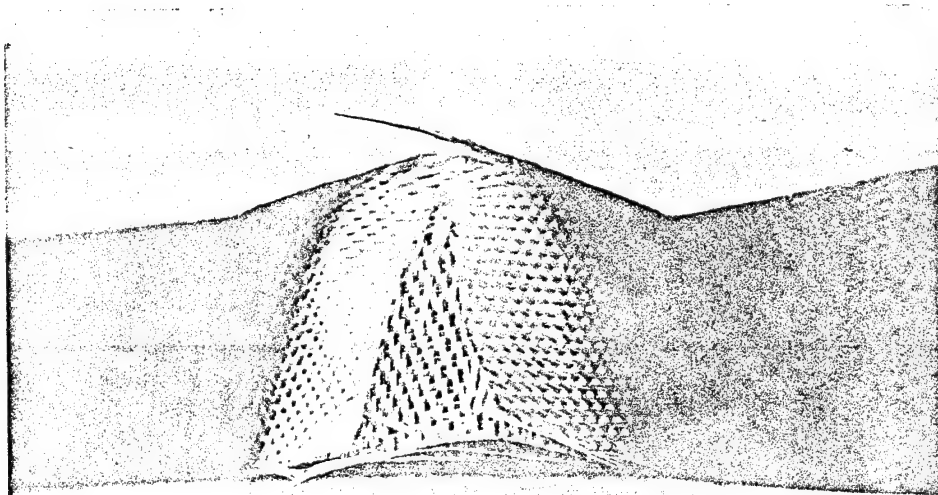
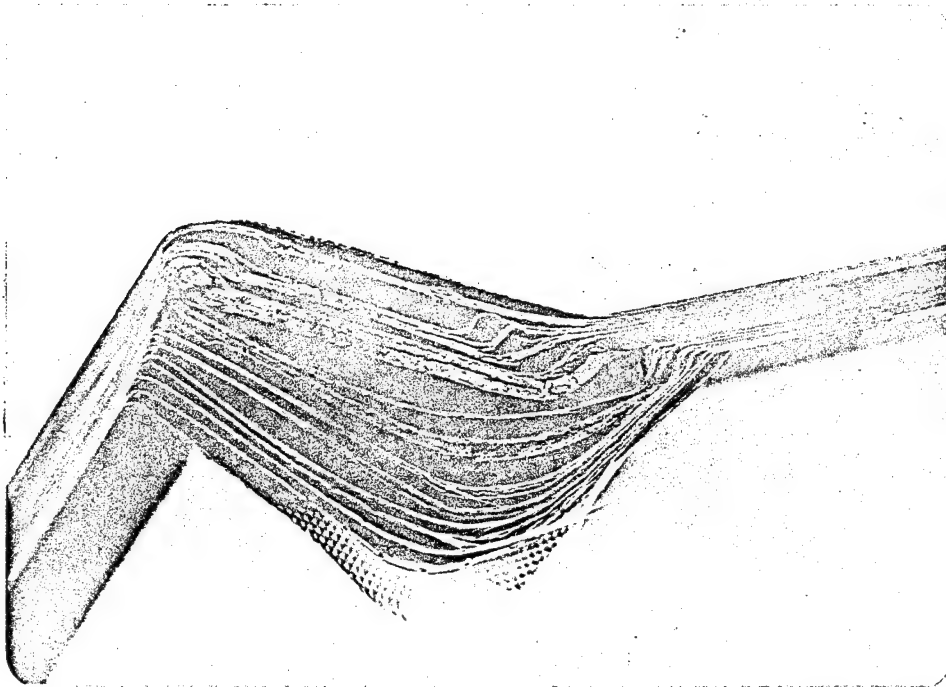
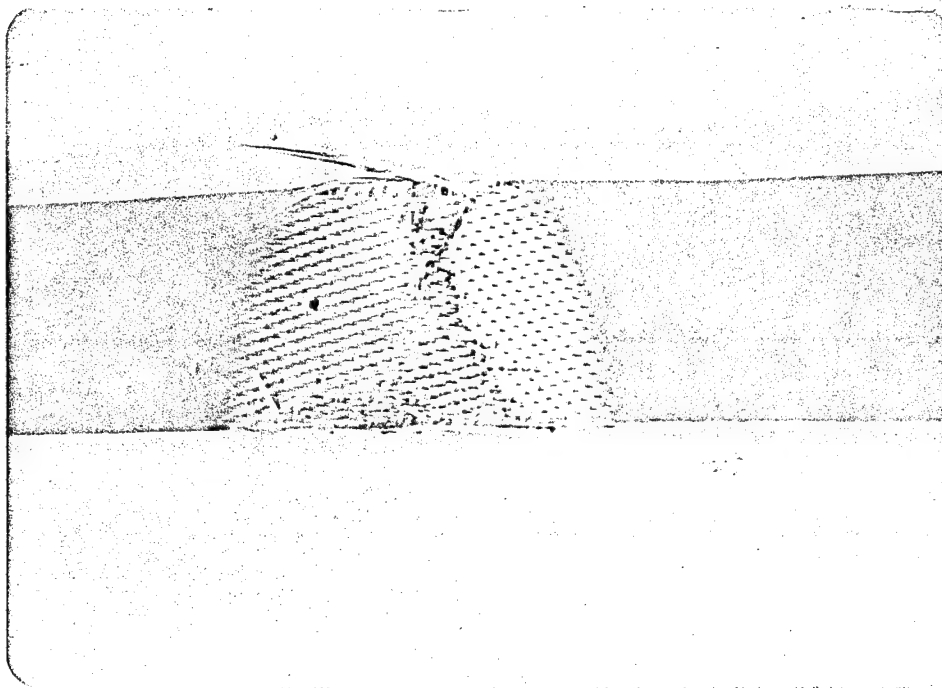
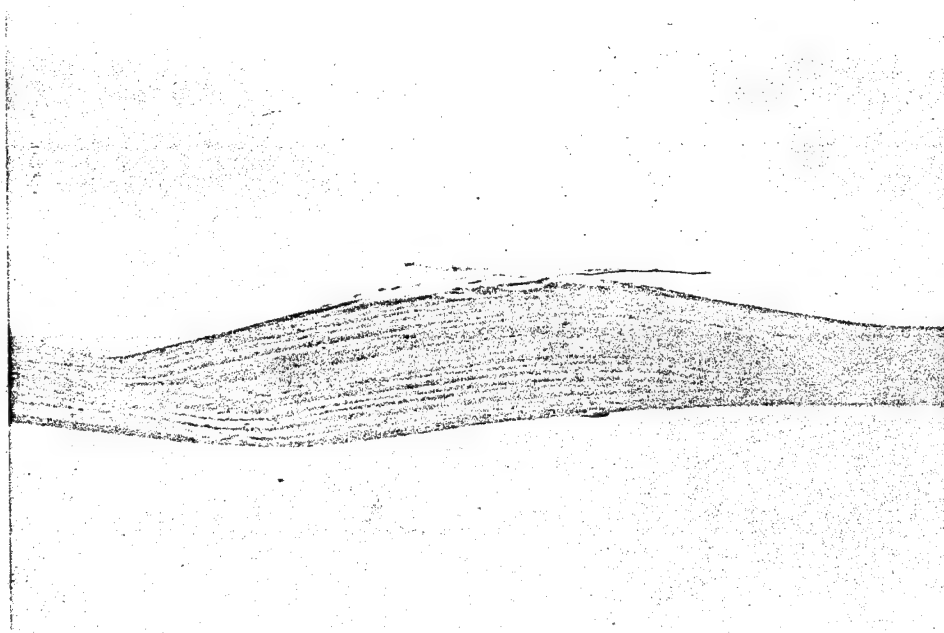


FIGURE 24. Sample from CD-24.
Example of minimum fiber release

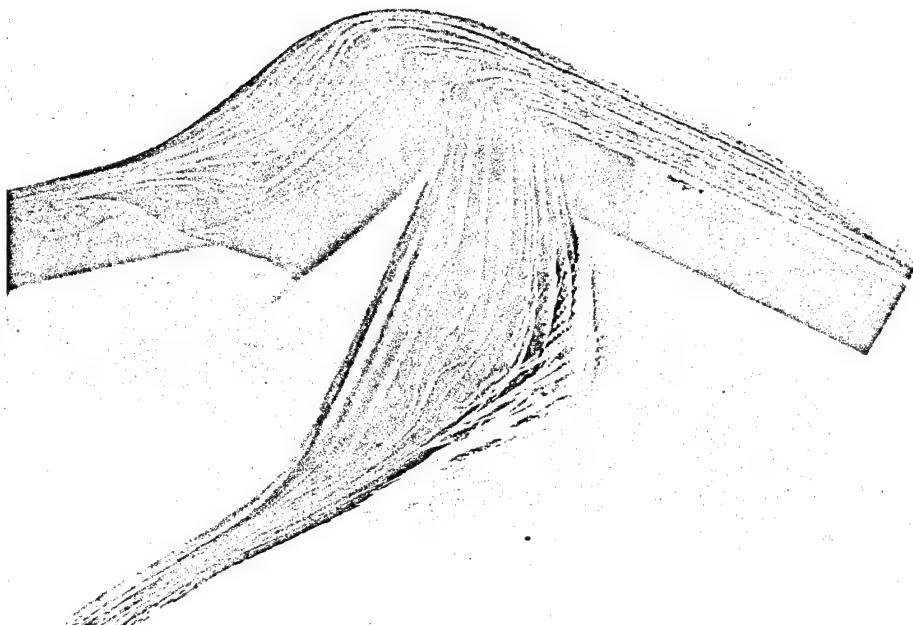


Front view (burn and impact testing)

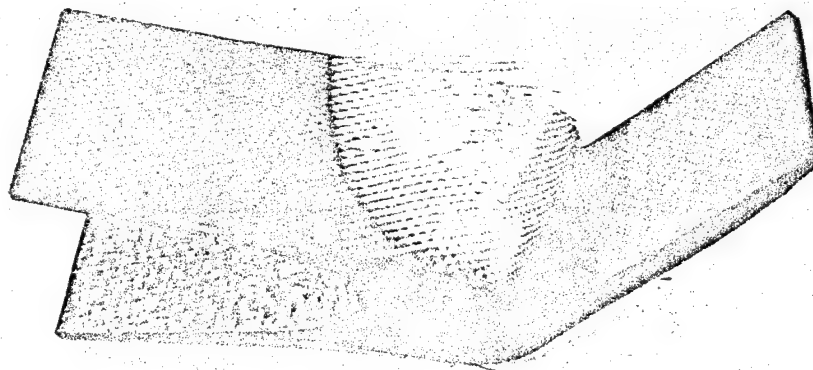


Side view above sample.

FIGURE 25. SAMPLE FROM CD-21.



Side view after testing



Front view from above sample

FIGURE 26. SAMPLE FROM CH-16.

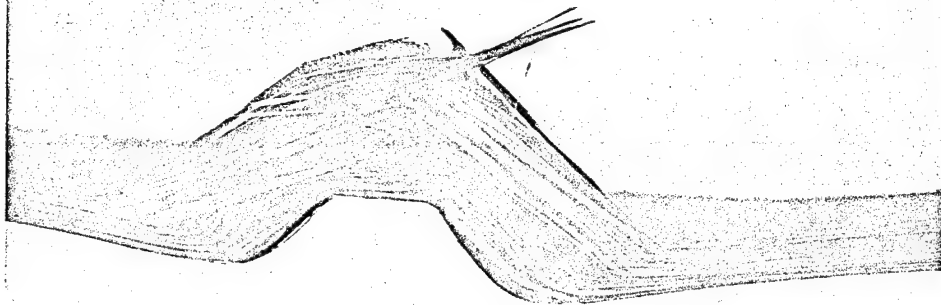


FIGURE 27. SIDE VIEW AFTER BURN-IMPACT TESTING DL-9.

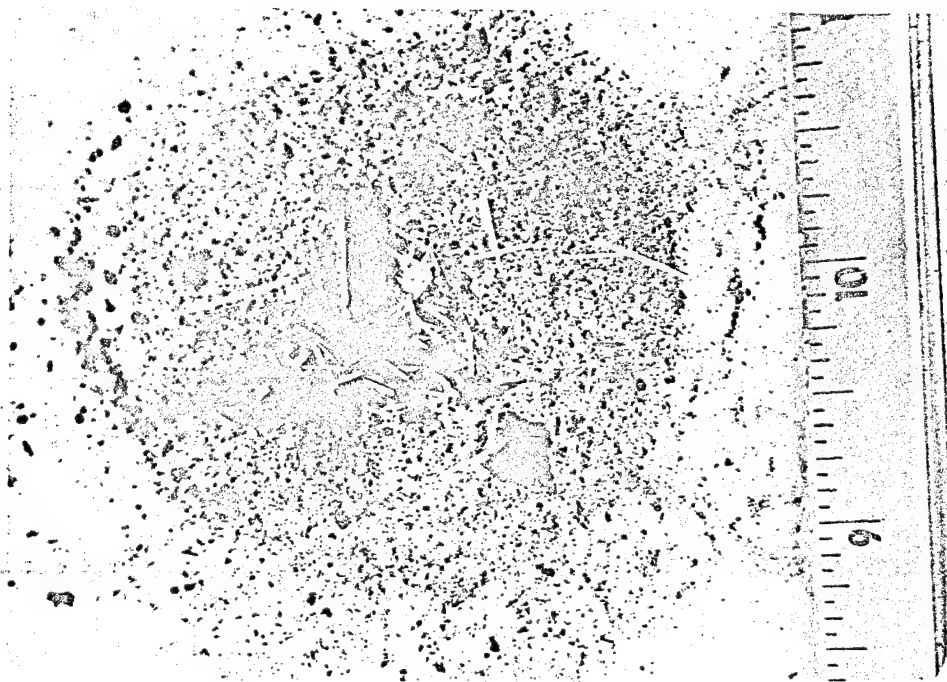
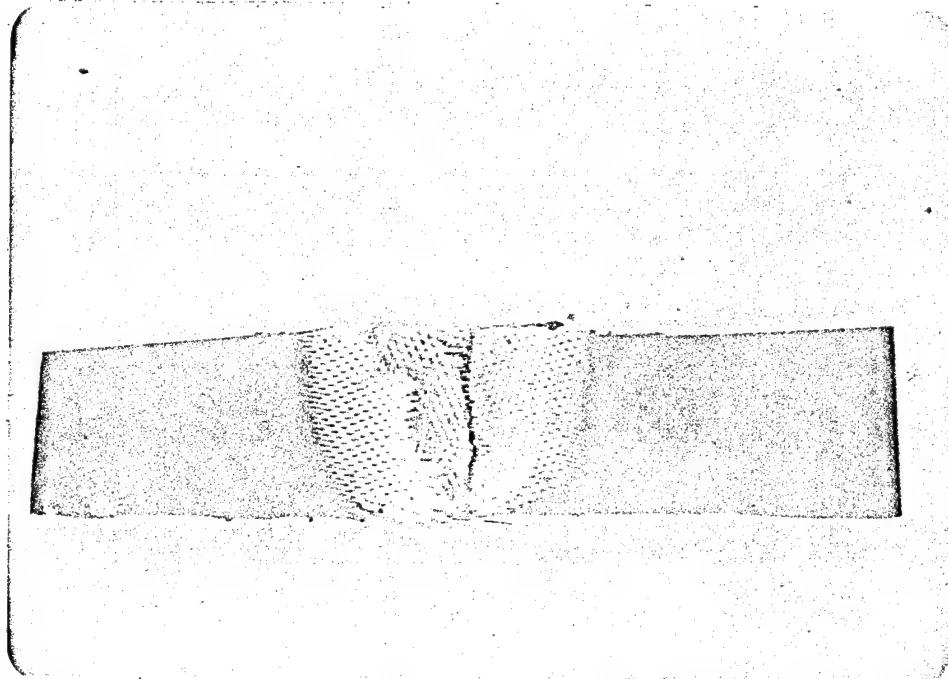
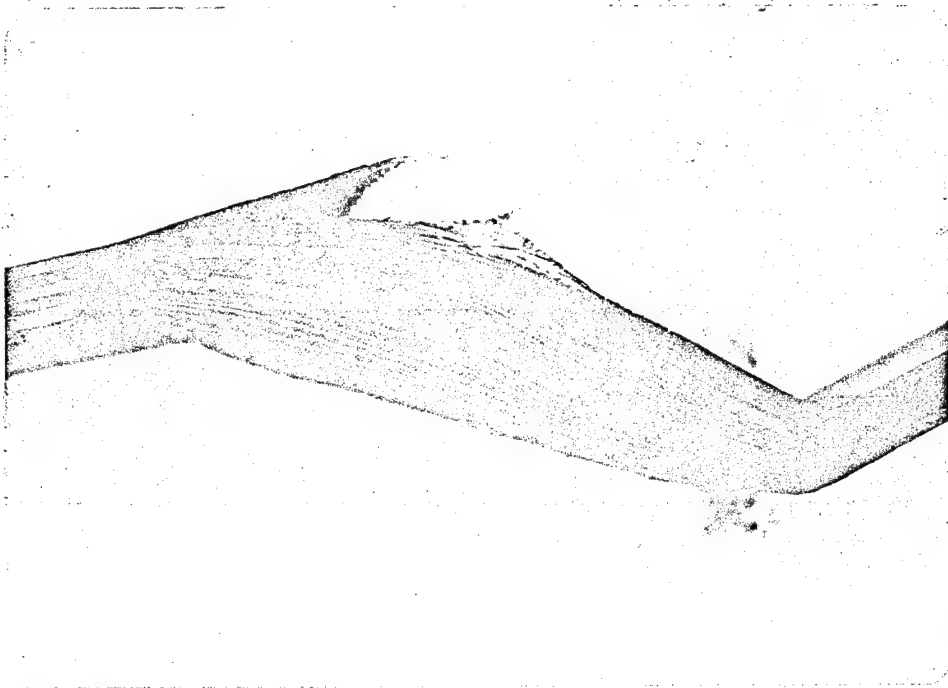
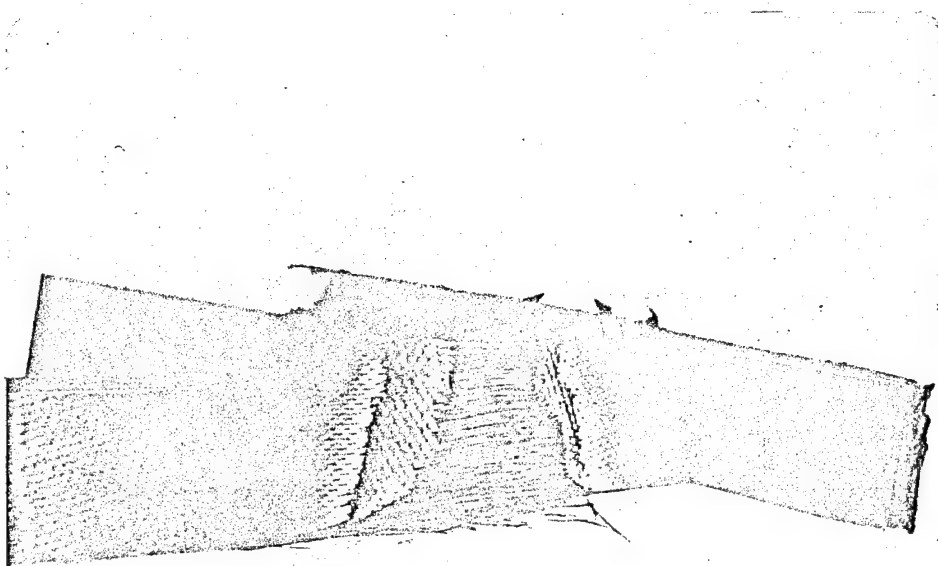


FIGURE 28. EJECTA FROM BOTTOM OF TESTER. DL-23 TEST.



Frontview and sideview after impact and burn test.

FIGURE 29. SAMPLE FROM CD-6.



Protective glass cover was burned away and some fibers released.

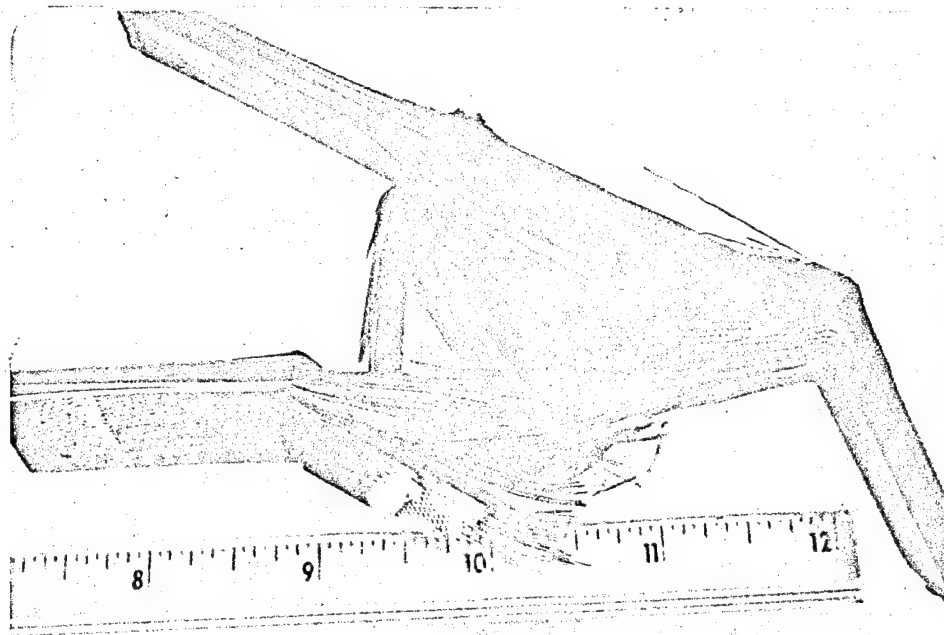


FIGURE 30. PANEL DL-3 SIDE VIEW AND FRONT VIEW AFTER BURN-IMPACT TESTING.

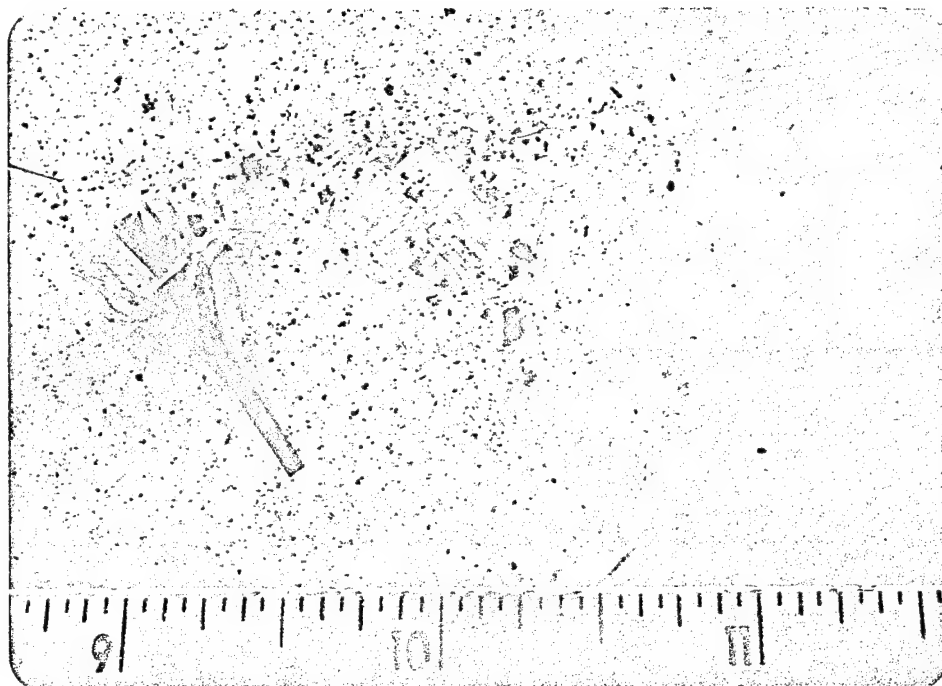


FIGURE 31. EJECTA FROM BOTTOM OF TESTER.
DL-3 TEST.

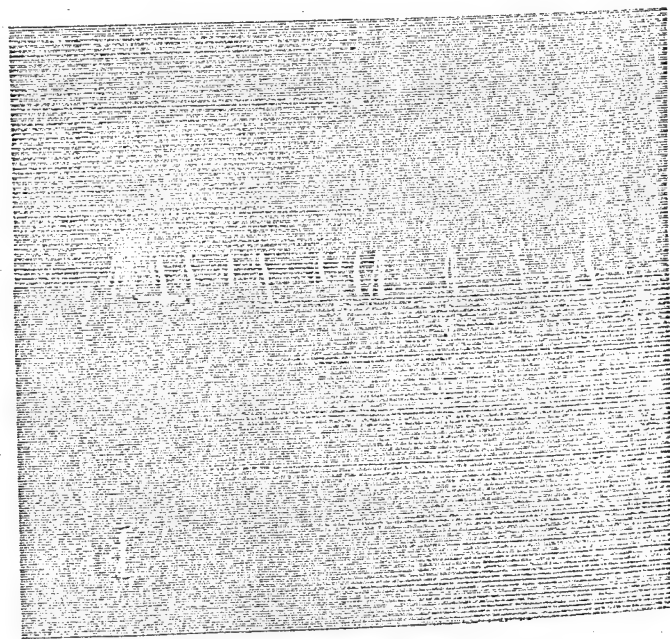
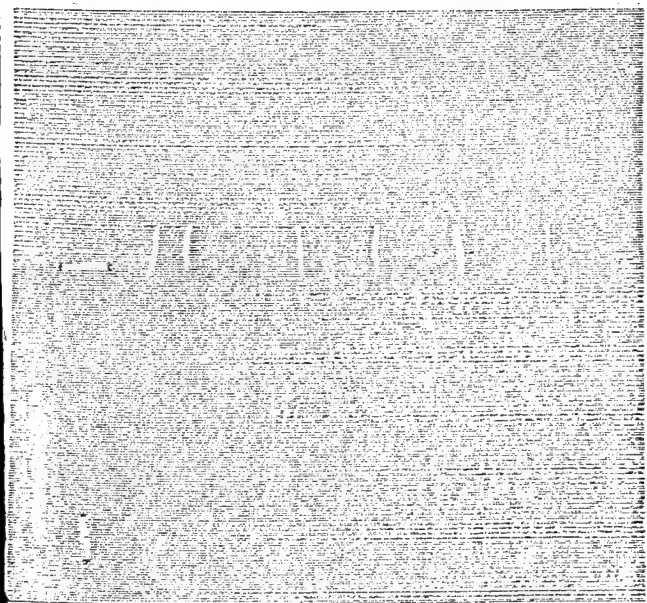
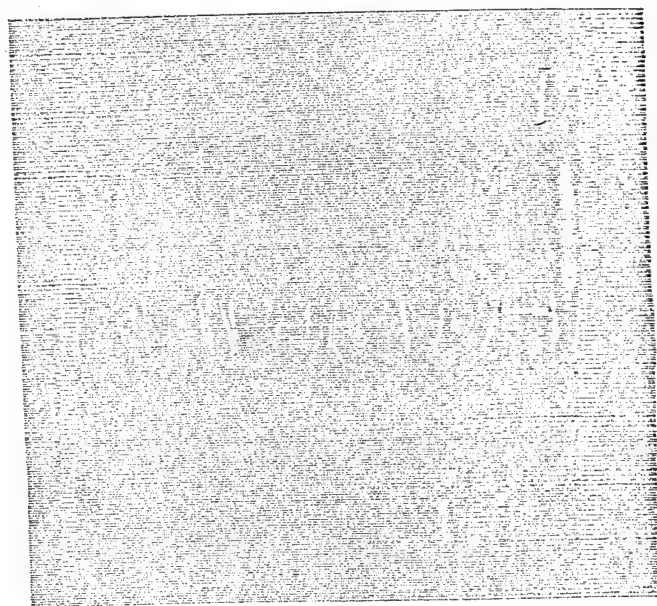
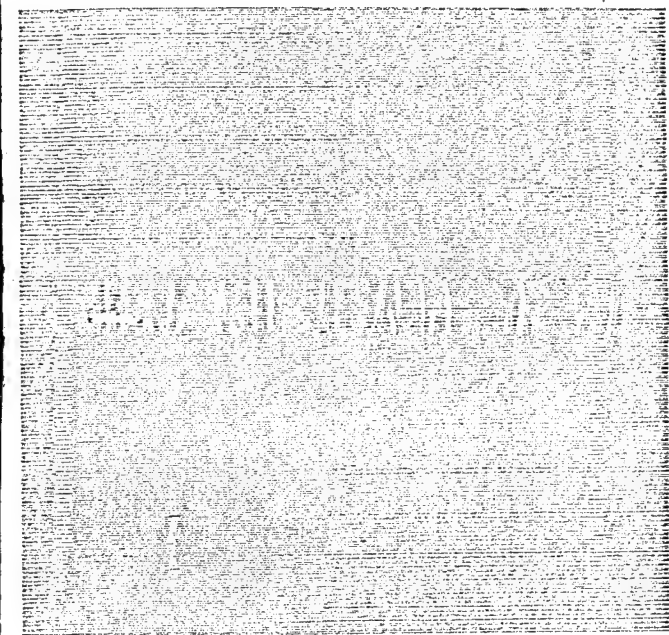


FIGURE 32. ULTRASONIC C SCAN RESULTS, FOUR PANELS

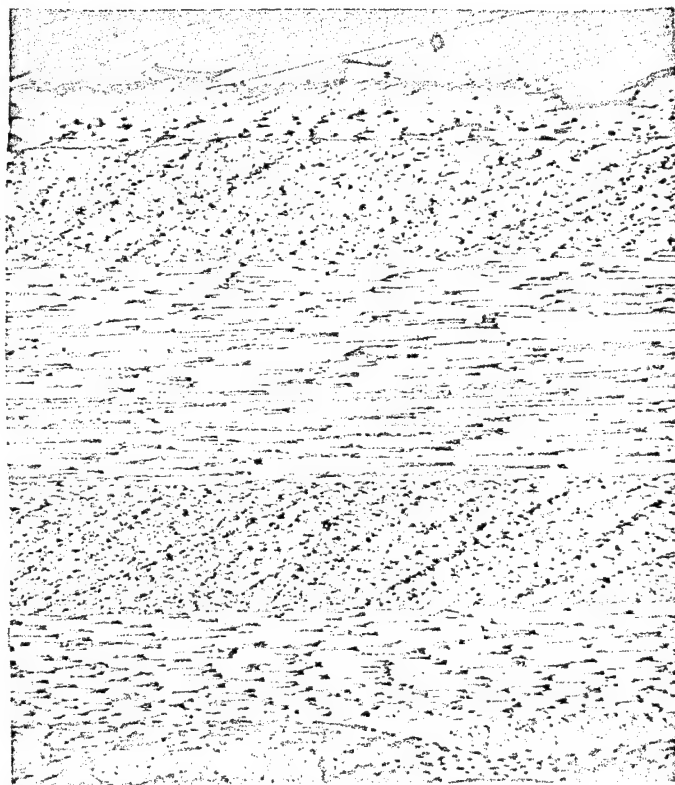


FIGURE 33. PHOTOMICROGRAPH
PANEL CH-8

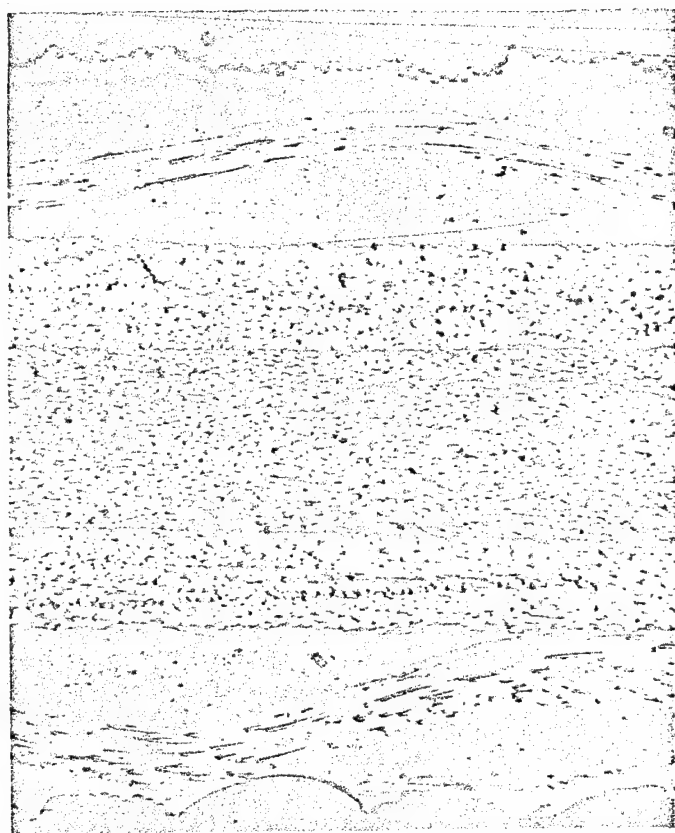
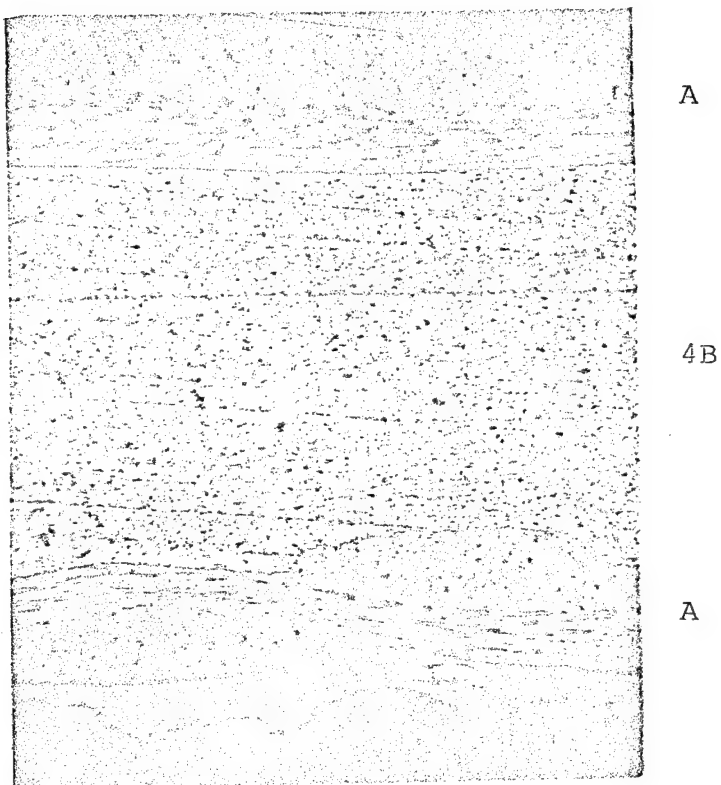
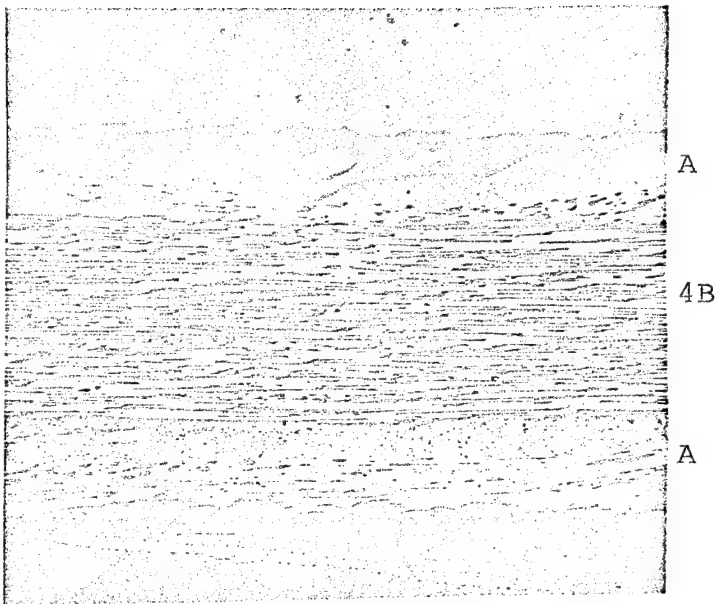


FIGURE 34. PHOTOMICROGRAPH
PANEL DL-18

FIGURE 35 . PHOTOMICROGRAPHS
OF CC-34 SAMPLES



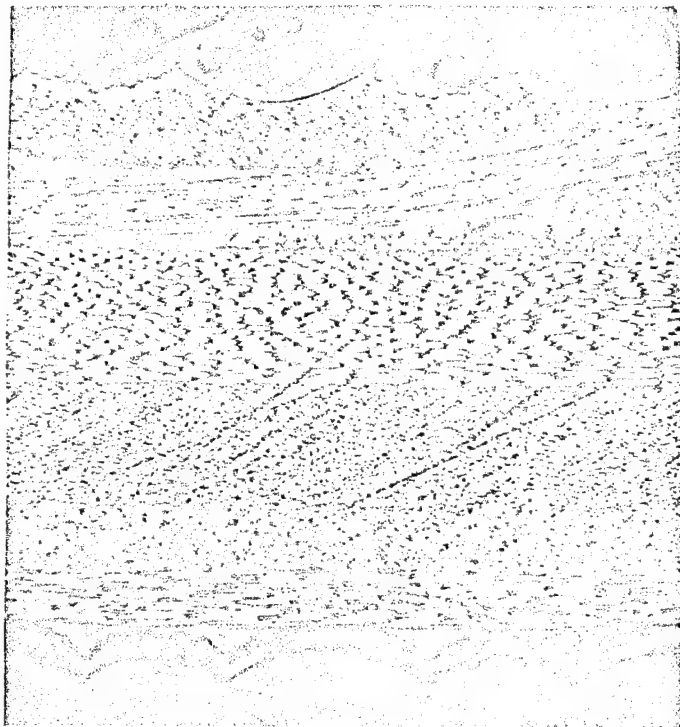


FIGURE 36. PHOTOMICRO-
GRAPH, PANEL CH-13

A

D

D (90)

A

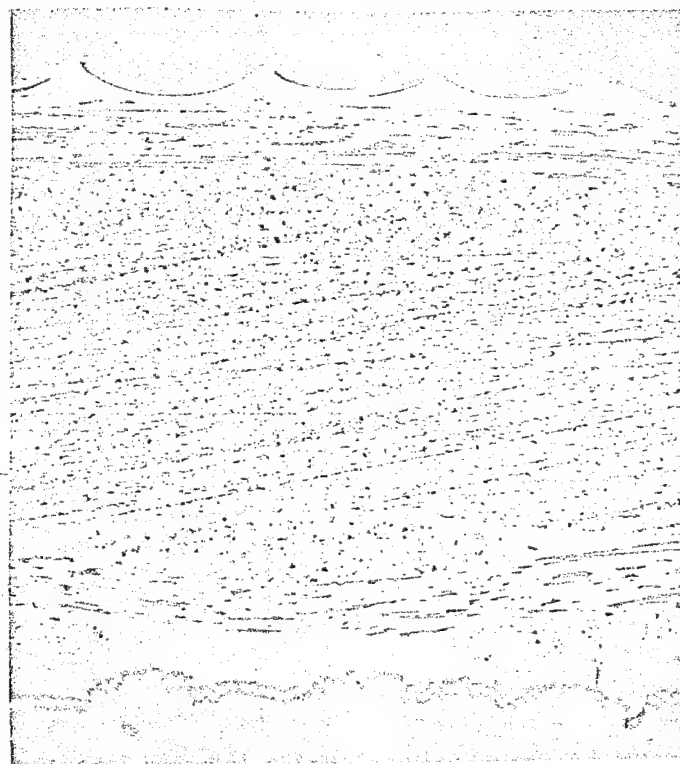


FIGURE 37. PHOTOMICRO-
GRAPH, PANEL CC-11

A

3B

A

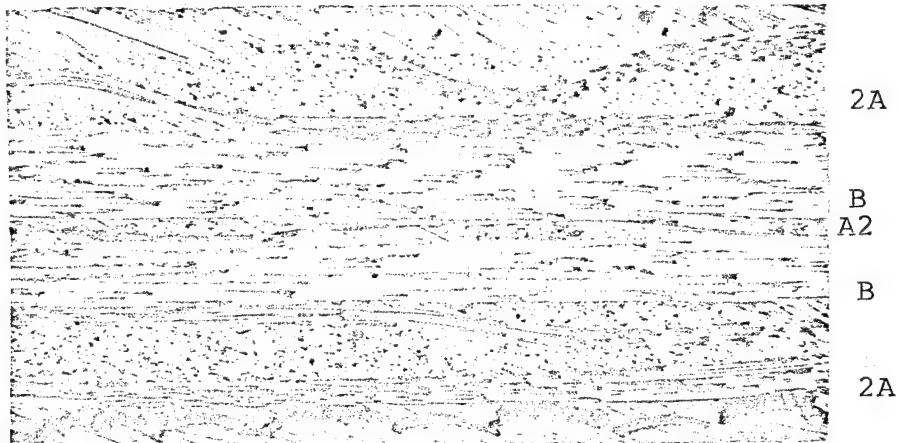


FIGURE 38. PHOTOMICROGRAPH, PANEL DL-27

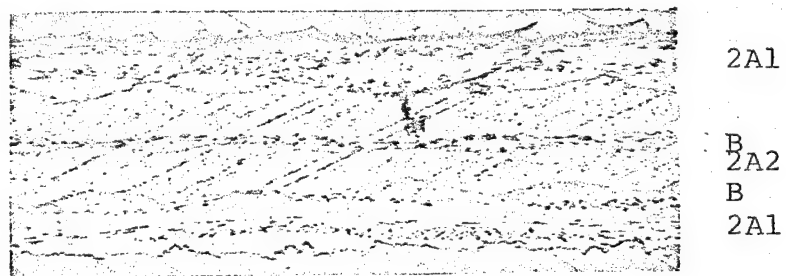


FIGURE 39. PHOTOMICROGRAPH PANEL CC-18

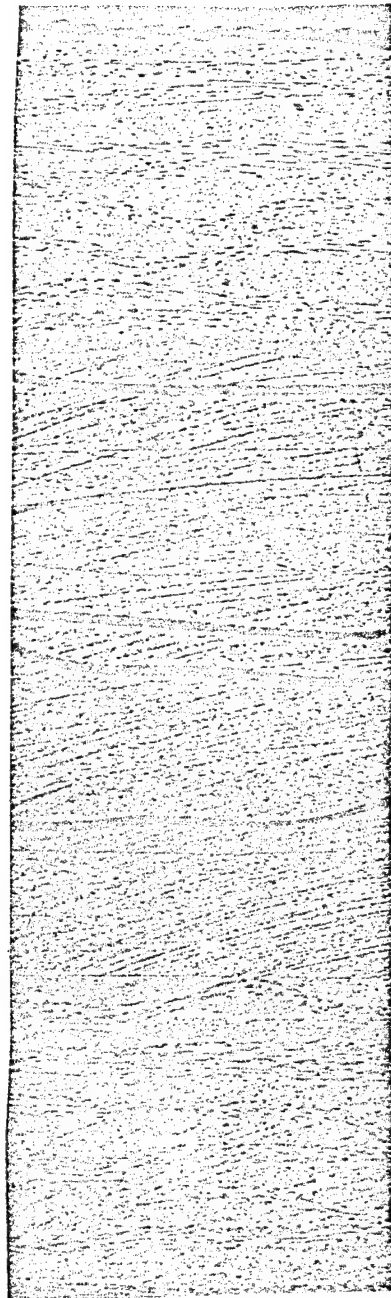


FIGURE 40. CH-10

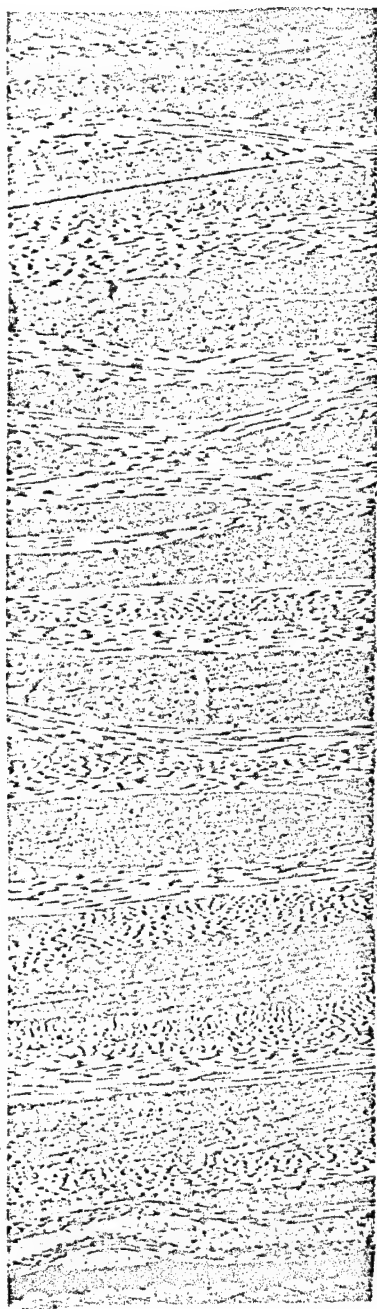


FIGURE 41.
CH 5

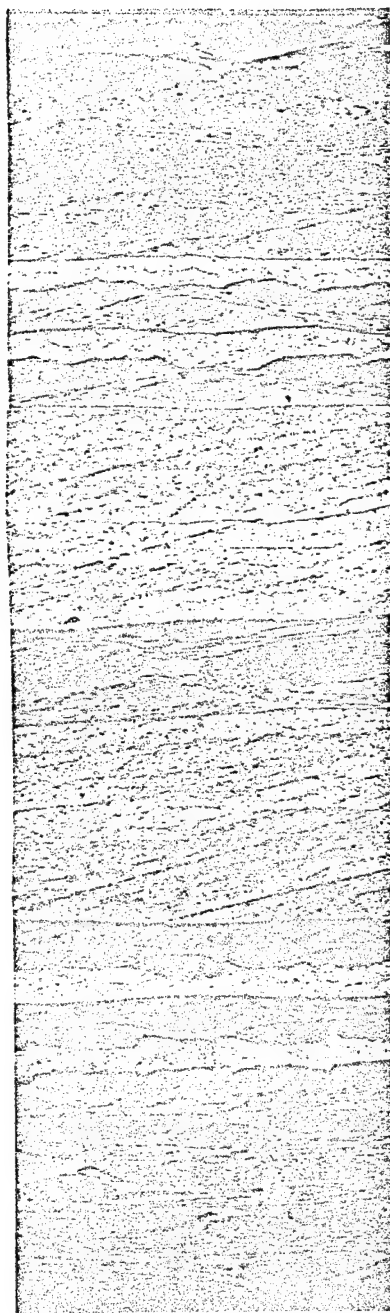


FIGURE 42.
CD 24

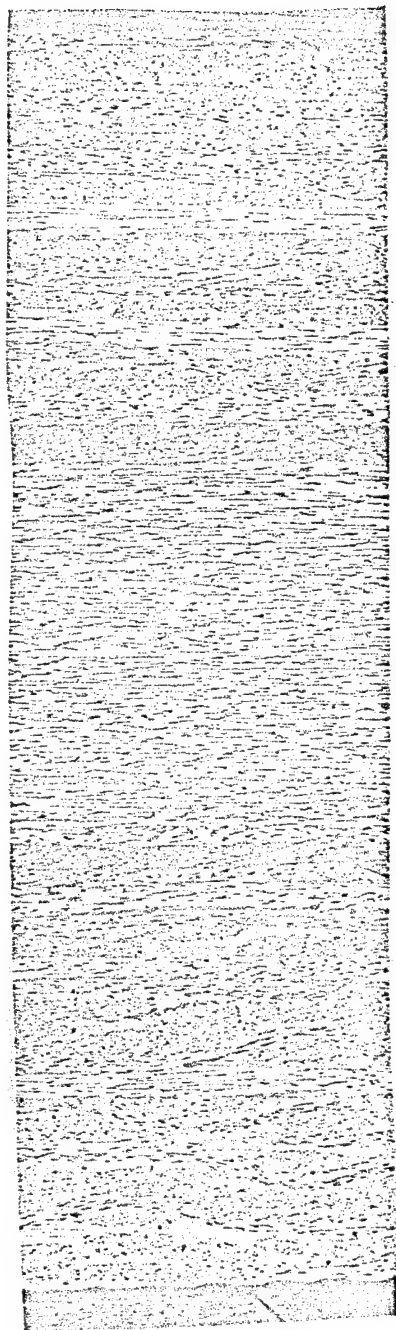


FIGURE 43.
CD-21

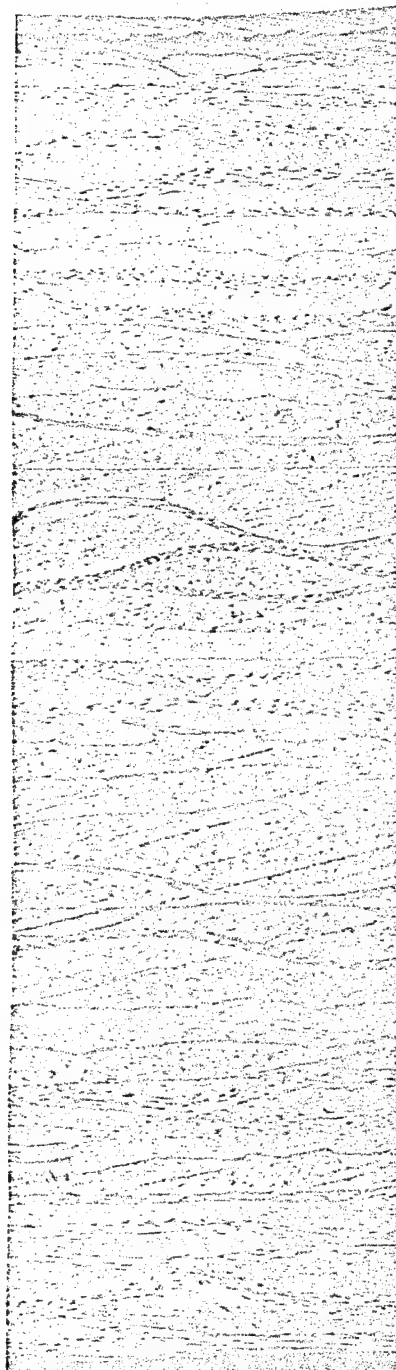


FIGURE 44.
CH-16

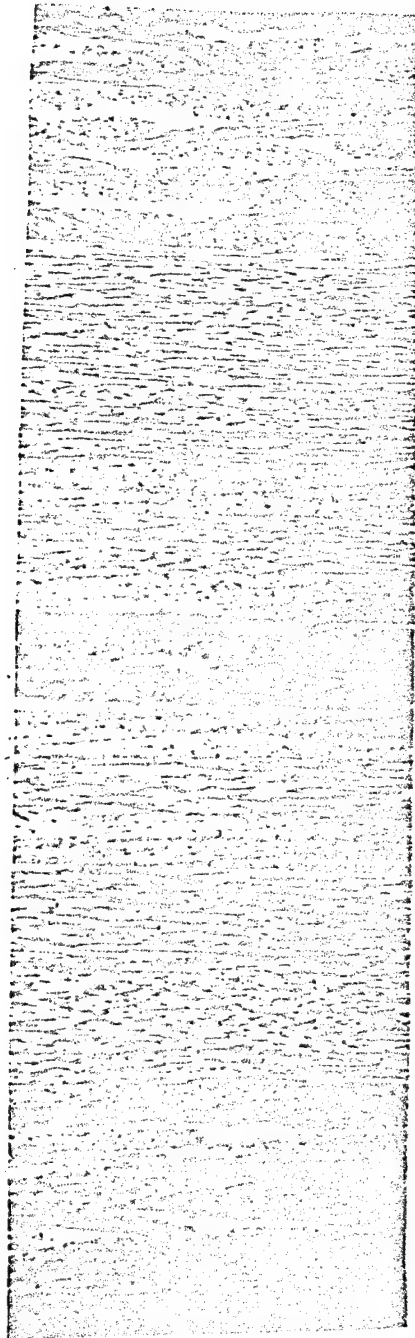


FIGURE 45. DL-9

TABLE 1.

SELECTION CRITERIA FOR PANEL CONFIGURATIONS

- 1) The stiffness of the composite must be comparable to that of aluminum (6.9×10^4 MPa)
- 2) Panels were studied in two different thicknesses:
 - a) between 0.64 to 1.02 mm (.025 - .040 in.), the "thin" panels
 - b) greater than 0.64 mm (.025 in.) the "thick" panels
- 3) Because glass does not burn, and adds to the strength of the structure, special emphasis was placed on glass/graphite hybrids.
- 4) Concentration on epoxy type resins due to wide use.
- 5) The structures should be as similar as possible to "real life" composites.
- 6) The structures should be practical with respect to production cost as well as raw material cost.

TABLE 2.

MATERIALS AND IDENTIFICATIONS

CODE FOR LETTERS USED IN TABLE 5.

A	E Glass cloth 0.0095 inches thick (Style 7781)
A ₁	E Glass Cloth 0.004 inches thick (Style 120)
A ₂	E Glass cloth 0.001 inches thick (Style 104)
B	Graphite unidirectional tape 0.005 inches thick
C	Graphite cloth 0.013 inches thick (W133)
D	Graphite glass cloth 0.007 inches thick (W190)
K	Phenol formaldehyde polymer cloth (Kynol)
K ₁	Kevlar cloth 0.010 inch thick (Style 281)

Example

A-B(45)-2B(-45)-B(45)-A

Represents:

- 1 ply E glass cloth (7781)
- 1 ply unidirectional graphite tape at +45°
- 2 plies unidirectional graphite tape at -45°
- 1 ply unidirectional graphite tape at +45°
- 1 ply E glass cloth (7781)

TABLE 3.

BURN/IMPACT TESTER COMPONENTS

- 1) High pressure air inlet valve (150 psi max)
- 2) Airfilter
- 3) Air pressure regulator
- 4) Air buffer tank (10 liters capacity)
- 5) Electrically operated air valve
(Norgren T41-DA-80, TSI, TDI)
- 6) Bronze air test gauge (Ashcroft, 4 1/2 inch)
- 7) Electrical switch
- 8) Clippard minimatic 18 D-6 air cylinder
- 9) Spherical tap (ballbearing, 1/2 inch diameter)
- 10) Sample panel in holder
- 11) Gas and pressurized air burner, generating heat
by radiation from ceramic cone (Duradant burner,
from Selas Corporation)
- 12) Polyester airfilter
- 13) Electrical fan
- 14) Optical pyrometer, range 200-1700°C (Barnes
Engineering Corporation)
- 15) Control unit for pyrometer
- 16) To propane bottle
- 17) Gas inlet valve
- 18) Pressurized air inlet valve

TABLE 4.

CONFIGURATION AND CALCULATED THICKNESS, THIN PANELS

ID	THICKNESS, MM(in.)	CONFIGURATION
CC-11	.864 (.034)	A-3B-A
CC-34	.991 (.039)	A-4B-A
DL-18	.991 (.039)	A-B(45)-2B(-45)-B(45)-A
DL-27	.686 (.027)	2A1-B-A2-B-2A1
CC-18	.711 (.028)	2A1-B-2A2-B-2A1
CC-36	.787 (.031)	A1-B-A2-B-A2-B-A2-B-A1
CH-8	.991 (.039)	A1-C-B-C-A1
CH-21	.838 (.033)	A-2D-A
CH-13	.838 (.033)	A-D-D(90)-A

TABLE 5.

CONFIGURATION AND CALCULATED THICKNESS, THICK PANELS

ID	THICKNESS, MM(in.)	CONFIGURATION
CD-5	6.40 (.252)	8A-20B-8A
CH-5	6.58 (.259)	2A-17C-2A
CH-10	6.71 (.264)	8A-16D-8A
CD-6	6.40 (.252)	6A-10B-4A-10B-6A
CD-11	6.40 (.252)	6A-2B-2A-16B-2A-2B-6A
CD-24	6.40 (.252)	5A-B-A-B-A-8B-A-B-A-B-5A
CD-21	6.40 (.252)	4A-B-2A-B-A-B-A-14B-A-B-A -B-2A-B-4A
CH-16	7.06 (.278)	4A-D-2A-D-A-7D-2A-7D-A-D -2A-D-4A
DL-9	6.45 (.254)	5A-8B-B(45)-B(45)-2B-B(-45)- B(45)-2A-B(45)-B(-45)-2B-B (45)-8B-5A
DL-23	7.24 (.285)	A-9D-D(45)-2D(-45)-2D(45) -2D(-45)-2D(45)-2D(-45)-2D (45)-2D(-45)-2D(45)-2D(-45)- -D(45)-9D-A
DL-3	6.55 (.258)	2A-10B-B(45)-2B(-45)-2B(45) -2B(-45)-2B(45)-2B(-45)-2B (45)-2B(-45)-2B(45)-2B(-45) -2B(45)-2B(-45)-B(45)-10B -2A

TABLE 6.

BURN/IMPACT DATA - COMPARISON OF ADDITIVE EFFECT

CONFIGURATION	ID ¹	mg ON FILTER	mg OF CARBON	mg ON BOTTOM	mg OF CARBON
A-D-D(90)-A	CH-13	0.9	0	100	0
	CH-13-U	5	1	160	6
6B(+45,90)S	EK-1	28	28	370	370
	EK-1-U	3.1	3	3 large pieces	

1. U designation refers to ulexite/boric acid additive used in panel construction

TABLE 7.

FLEXURAL TEST RESULTS WITH AND WITHOUT ADDITIVE

CONFIGURATION	ID	FLEX STRENGTH MPa (KSI)	FLEX MODULUS GPa (MSI)	TEST TEMP (°F)	NOTES
2A1-B-2A2-B-2A1	CC18	593 (86)	10.3 (1.5)	RT	1
	CC18-U	299 (43)	7.9 (1.15)	RT	2
A-2D-A	CH13	490 (71)	29 (4.2)	RT	
	CH13	620 (90)	39 (5.7)	450 (350)	
	CH13U	731 (106)	28 (4.0)	RT	3
	CH13U	620 (90)	33 (4.8)	450 (350)	

Notes:

- 1) Early panel, later data in Tables 14 and 15
- 2) Additive not finely ground - preliminary test
- 3) Finely ground additive (37 micron maximum grain size)

TABLE 8.

CALCULATED PROPERTIES OF
CANDIDATE CONFIGURATIONS-THIN PANELS

ID	E_1 GPa (MSI)	E_2 GPa (MSI)	G_{12} GPa (MSI)	ν_{12}	ν_{12}
CC-11	79.3 (11.5)	19.0 (2.75)	6.0 (0.87)	0.17	0.042
CC-34	87.5 (12.7)	17.9 (2.60)	5.8 (0.84)	0.18	0.037
DL-18	25.0 (3.63)	24.1 (3.49)	22.9 (3.32)	0.57	0.55
DL-27	71.0 (10.3)	19.9 (2.89)	6.3 (0.91)	0.17	0.048
CC-18	69.6 (10.1)	20.1 (2.92)	6.2 (0.90)	0.17	0.05
CC-36	103.4 (15.0)	16.1 (2.33)	5.4 (0.79)	0.19	0.029
CH-8	76.5 (11.1)	58.7 (8.51)	0.5 (0.07)	0.047	0.036
CH-21	71.0 (10.3)	20.6 (2.99)	6.1 (0.88)	0.16	0.047
CH-13	46.9 (6.81)	45.4 (6.58)	6.1 (0.88)	0.074	0.072

TABLE 9.

CALCULATED PROPERTIES OF CANDIDATE CONFIGURATIONS

THICK PANELS

ID	<u>E₁</u>	<u>E₂</u>	<u>G₁₂</u>	<u>v₁₂</u>	<u>v₂₁</u>
	GPa (MSI)	GPa (MSI)	GPa (MSI)		
CD5	73.8 (10.7)	19.6 (2.84)	6.1 (0.88)	0.17	0.04
CH5	71.0 (10.3)	71.0 (10.3)	4.9 (0.71)	0.13	0.049
CH10	71.7 (10.4)	20.6 (2.99)	6.0 (0.87)	0.16	0.047
CD6	73.8 (10.7)	19.6 (2.84)	6.1 (0.88)	0.17	0.046
CD11	73.8 (10.7)	19.6 (2.84)	6.1 (0.88)	0.17	0.046
CD24	73.8 (10.7)	19.6 (2.84)	6.1 (0.88)	0.17	0.046
CD21	73.8 (10.7)	19.6 (2.84)	6.1 (0.88)	0.17	0.046
CH16	74.5 (10.8)	20.3 (2.95)	6.0 (0.87)	0.16	0.044
DL9	73.8 (10.7)	21.8 (3.17)	8.2 (1.19)	0.37	0.11

TABLE 10

TEST DATA ON COMPOSITE STRUCTURES

BURN/IMPACT TESTING
THIN PANELS

CONFIGURATION	mg ON FILTER	mg OF CARBON	mg ON BOTTOM	mg OF CARBON	I.D.
6B(+45,90) _S	28	28	376	376	EK-1
Al-C-B-C-Al	0	0	716	20	CH-8
A-B(45)-2B(-45)-B (45)-A	1.9	1.0	524	524	DL-18
Al-B-A2-B-A2-B-A2 -B-Al	1.0	1.0	0	0	CC-36
	0.3	0.1	54	5	
A-2D-A	0.9	0	100	0	CH-21
A-3B-A	2.6	2.6	NA	NA	CC-11
	57	57	437	437	
	3.2	3	63	6	
	2	2	283	200	
	2.1	2.1	0	0	
A-4B-A	4.8	4.8	140	84	CC-34
	390	390	475	400	
	1.4	1.4	137	30	
	6.5	6.5	137	NA	
2Al-B-A2-B-2Al	1.0	1.0	207	NA	DL27
	2.1	1.0	261	20	

TABLE 11

TEST DATA ON COMPOSITE STRUCTURES
BURN/IMPACT TESTING THICK PANELS

CONFIGURATION	mg ON FILTER	mg OF CARBON	mg ON BOTTOM	mg OF CARBON	I.D.
8A-20B-8A	0	0	2470	1	CD-5
	5	5	92	50	
8A-16D-8A	9	0	15	trace	CH-10
	9	3	NA	NA	
2A-17C-2A	0.7	0.7	136	trace	CH-5
6A-2B-2A-16B-2A- 2B-6A	7.4	NA	819.5	NA	CD-11
	4.2	NA	309	150	
5A-B-A-B-A-8B- 2A-8B-A-B-A-B-5A	2.8	2.8	21	10	CD-24
	0.7	0.7	272	very little	
4A-B-2A-B-A-B-A- 14B-A-B-A-B-2A- B-4A	8.4	2	37	18	CD-21
	8.4	7	1660	about 20	
4A-D-2A-D-A-7D- 2A-7D-A-D-2A-D-4A	2.8	2.8	28	28	CH-16
5A-8B-B(45)-B(-45)- 2B-B(-45)-B(45)-2A -B(45)-B(-45)-2B- B(-45)-B(45)-8B-5A	1.4	1.4	28	very little	DL-9
1A-9D-D(45)-2D(-45) -2D(45)-2D(-45)-2D (45)-2D(-45)-2D(45) -2D(-45)-2D(45)-2D (-45)-D(45)-9D-1A	23	5.4	390	390	DL-23
6A-10B-4A-10B-6A	6.6	6.0	83	trace	CD-6
	6.3	6.3	167	very little	
2A-10B-B(45)-2B(-45) -2B(45)-2B(-45)-2B (45)-2B(-45)-2B(45) -2B(-45)-2B(45)-2B (-45)-2B(45)-2B(-45) -B(45)-10B-2A	4.2	4.2	48	about half	DL-3
	14	14	309	about half	

NOTES ON TABLES 10 AND 11 OF THE BURN/IMPACT TEST RESULTS

ID EK-1

Analysis:

Air filter residue very heavy, with many long fibers. Burn temperature was 670°C. The panel nearly fell apart (see Figure 10). This test was repeated many times on different panels, with similar results.

Conclusion:

Panel failed. Typical behavior of unprotected panel.

ID CH-8

Analysis:

Burn temperature was 610°C. Very little destruction observed (see Figure 18).

Conclusion:

An excellent configuration.

ID DL-18

Analysis:

Burn temperature was 580°C-660°C. Considerable amounts of single strand fibers were found on the bottom of the tester (see Figure 21), which would be easily transportable in a faster air stream. Many of the 45° plies are exposed in the 25.4 mm (1 in.) wide sample, as the resin has been burned away over their total length.

Conclusion:

Not acceptable as tested, possibly due to edge effect.

ID CC-36

: Analysis:

The two panels tested differed in their burning temperature. The first sample was burned at 850°C. The second at 650°C-700°C. Considerable quantity of free fibers at the panel edges.

Conclusion:

Acceptable.

NOTES ON TABLES 10 AND 11 (CONT.)

ID CH-21

Analysis:

Burn temperature between 640°C-700°C.

Conclusion:

Panel acceptable.

ID CC-11

Analysis:

The burn-impact testing on this structure was repeated many times. The 2.1 mg carbon on the air filter came from a panel of 76.2X101.6 mm size, which filled the whole front of the sample holder (see Figure 8).

The sample with the 2.0 mg fibers on the air filter resulted from a trial in which the burn temperature was in excess of 700°C. The fibers were all smaller than 2mm, but the panel was burned into two pieces.

At 660°C burn temperature there was 57 mg on the filter, which made the structure unacceptable, but structures like A-3B-A and A-4B-a were considered as there were not enough combinations of materials in these thicknesses for structures between 0.64 to 1.02 mm thicknesses.

ID CC-34

Analysis:

One sample gave 390 mg carbon on impact. In this case the fibers were oriented at right angles to the long edge of the 25.4 mm wide panel and many were unsupported after the burn. The tup destroyed the panel on impact (see Figure 19).

ID DL-27 and CH-18

Analysis:

The structures behaved very similarly and very acceptably. It seemed that for the thin panels an intimate mixture of glass and graphite was the most resistant to these testing conditions. (See Figure 21.)

NOTES ON TABLES 10 AND 11 (CONT.)

ID CD-5

Analysis:

The first sample (with 0 mg on air filter) had a lower burning temperature (645°C), yet all the resin over a length of 9 cm was burned away. The burning temperature at the second sample was over 700°C (see Figures 15, 16 and 17).

Conclusion:

Acceptable.

ID CH-10

Analysis:

There were some very long carbon fibers found (see Figure 22).

Conclusion:

Structure is acceptable.

ID CH-5

Analysis:

This structure was resistant to heating above 700°C and subsequent impact (see Figure 23). Only 3 plies of glass were burned, and no graphite.

Conclusion:

Acceptable.

ID CD-11

Analysis:

Completely similar results were obtained with this structure as with CD-6.

Conclusion:

This structure is acceptable.

ID CD-24

Analysis:

Again the difference between the two samples was in their burning temperature. The first was 650°C, (see Figure 24) the latter 760°C.

Conclusion:

Acceptable.

NOTES ON TABLES 10 AND 11 (CONT.)

ID CD-21

Analysis:

Burning temperature for the first sample was 700°C (see Figure 25), for the second 780°C. The results were very similar.

Conclusion:

Structure is acceptable.

ID CH-16

Analysis:

Panel folded badly on impact (see Figure 26), but fiber release was minimal.

Conclusion:

Structure acceptable.

ID DL-9

Analysis:

Burning temperature was 720°C (see Figure 29). There were some fibers sticking out at the edges.

Conclusion:

Acceptable.

ID DL-23

Analysis:

Burn temperature of the first sample was 710°C, the second 880°C. Panel started to fall apart with a large quantity of carbon fibers released. A second hit with the impact tester gave 700 mg fibers (see Figure 28).

Conclusion:

Structure is unacceptable.

NOTES ON TABLES 10 AND 11 (CONT.)

ID CD-6

Analysis:

The sample with the most carbon on the air filter (6.6 mg) was burned at 660°C (see Figure 29), the other sample at 802°C.

Conclusion:

This configuration is acceptable.

ID DL-3

Analysis:

It was shown through previous experiments, that panels built with the glass-graphite cloth were very resistant. The panel was therefore burned at 900°C, which burned the protective glass cloth totally away. The impact fluffed 60% of all the plies up, but only 5 mg of fibers were found on the air filter. Nearly all the fibers on the tester bottom were contained as tows, not as single strand fibers (see Figures 30 & 31).

Conclusion:

Might be acceptable.

TABLE 12

ANALYTICAL RESULTS FROM ACID DIGESTION

I.D.	NUMBER OF SAMPLES	SPECIFIC GRAVITY (St Dev)	RESIN CONTENT (Wt%) (St Dev)	FIBER VOLUME (%) (St Dev)	VOID CONTENT (Vol%) (St Dev)
CC-11	3	1.7880 (0.00057)	30.7 (0.15)	56.9 (0.11)	0.1 (0.11)
CC-34	3	1.749 (0.0093)	33.1 (0.83)	54.2 (0.05)	0.4 (0.17)
CC-18	3	1.773 (0.0050)	32.5 (0.17)	54.66 (0.043)	0.2 (0.38)
DL-27	3	1.79 (0.017)	31.7 (0.37)	55.2 (0.83)	0.3 (0.74)
CD-5	3	1.794 (0.002)	31.6 (0.002)	54.7 (0.23)	0.72 (0.080)
CD-6	3	1.816 (0.0019)	30 (1.6)	56 (1.3)	0.1 (1.0)
CD-11	3	1.8121 (0.00057)	29.2 (0.67)	47.2 (0.54)	1.1 (0.42)
CD-11	3	1.836 (0.0013)	27.5 (0.22)	59.4 (0.23)	0.9 (0.13)
CD-24	3	1.824 (0.0026)	29.3 (.34)	57.61 (0.36)	0.47 (0.084)
CD-21	3	1.791 (0.0026)	31.4 (0.39)	54.9 (0.39)	1.0 (0.13)

TABLE 13.
TEST RESULTS - SHORT BEAM SHEAR STRENGTH

I.D.	ROOM TEMPERATURE			TEMPERATURE 450°K		
	No. of Samples	Average Ultimate Stress MPa (KSI)	St.Dev MPa	No. of Samples	Average Ultimate Stress MPa (KSI)	St Dev MPa
<u>THIN PANELS</u>						
CC-11	5	61 (8.8)	4.0	5	36 (5.2)	2.0
CC-34	5	76 (11.0)	4.5	5	40 (5.8)	2.5
CH -13	5	43 (6.2)	4.5	5	25 (3.6)	1.8
DL-18	5	50 (7.3)	3.5	5	30 (4.4)	1.0
CH -8	5	59 (8.6)	1.6	5	26 (3.8)	1.2
DL-27	5	34 (4.9)	1.2	5	29 (4.2)	1.2
CC-18	5	45 (6.5)	2.2	5	33 (4.8)	2.7
<u>THICK PANELS</u>						
CD-5	6	80 (11.6)	9	6	44 (6.4)	2.5
CH -10	3	52 (7.5)	4.2	3	27 (3.9)	1.3
CD-6	6	68 (9.9)	1.1	6	45 (6.5)	2.1
CD-24	6	60 (8.7)	4.3	5	50 (7.3)	2.0
CH -16	4	50 (7.3)	1.4	4	32 (4.6)	1.7
DL-9	4	68 (9.9)	3.2	4	46 (6.7)	3.6
CD-21	6	76 (11.0)	3.1	6	44 (6.4)	2.7
CH-5	3	28 (4.1)	1.4	3	23 (3.3)	3.0
CD-11	5	78 (11.3)	3.0	6	46 (6.7)	1.6
CD-11	6	79 (11.5)	4.1	6	45 (6.5)	4.0
CD-11	6	77 (11.2)	1.2	6	43 (6.2)	1.4
CD-11	6	60 (8.7)	1.0	6	45 (6.5)	4.7

TABLE 14.

TEST RESULTS-FLEXURAL STRENGTH

I.D.	ROOM TEMPERATURE			TEMPERATURE 450°K		
	No. of Samples	Average Ultimate Stress MPa (KSI)	St.Dev MPa	No. of Samples	Average Ultimate Stress MPa (KSI)	St Dev MPa

THIN PANELS

CC-11	3	880 (128)	56	3	830 (120)	76
CC-34	3	1000 (145)	11	3	900 (130)	44
CH-21	3	490 (71)	63	3	620 (90)	75
DL-18	3	850 (123)	46	3	660 (96)	11
DL-27	3	560 (81)	55	3	410 (59)	82
CC-18	3	770 (112)	48	3	680 (99)	67

THICK PANELS

CD-5	3	720 (104)	10	3	577 (84)	1.6
CH-5	3	320 (46)	53			
CH-10	2	552 (80)	3.6	2	361 (52)	1.3
CD-6	3	972 (141)	9	3	760 (110)	91
CD-21	3	810 (117)	28	3	650 (94)	19
CD-24	3	850 (123)	32	3	725 (105)	10
CH-16	3	510 (74)	27			
DL-9	2	1257 (182)	5	2	790 (115)	16
CD-11	3	800 (116)	24	3	680 (99)	21
CD-11	2	810 (117)	48	2	550 (80)	83

TABLE 15.

TEST RESULTS-FLEXURAL MODULUS

I.D.	No. of Samples	ROOM TEMPERATURE		No. of Samples	TEMPERATURE 450°K	
		Average Ultimate Stress MPa (KSI)	St.Dev MPa		Average Ultimate Stress MPa (KSI)	St Dev MPa

THIN PANELS

CC-11	3	33 (4.8)	2.7	3	23 (3.3)	1.4
CC-34	3	36 (5.2)	3.3	3	32 (4.6)	1.8
CH-21	3	29 (4.2)	1.0	3	39 (5.7)	4.6
DL-18	3	25 (3.6)	1.0	3	31 (4.5)	2.3
CH-8	3	36 (5.2)	2.4	3	33 (4.8)	0.8
DL-27	3	23 (3.3)	0.9	3	18 (2.6)	1.2
CC-18	3	34 (4.9)	7.6	3	26 (3.8)	3.7

THICK PANELS

CD-5	3	32 (4.6)	0.8	3	31 (4.5)	1.5
CH-5	3	48 (7.0)	1.4			
CH-10	2	25.3 (3.7)	0.01	2	21.2 (3.1)	0.02
CD-6	3	41.8 (6.1)	0.3	3	37 (5.4)	2.4
CD-24	3	37 (5.4)	1.8	3	36.5 (5.3)	0.8
CD-21	3	45 (6.5)	1.4	3	34.2 (5.0)	0.1
CH-16	3	25 (3.6)	1.0			
DL-9	2	49 (7.1)	2.1	2	49 (7.1)	0.2
CD-11	3	36.8 (5.3)	0.4	3	34.7 (5.0)	0.6
CD-11	2	36.1 (5.2)	0.5	2	29 (4.2)	5.4

TABLE 16.

CONFIGURATIONS OF PANELS DELIVERED TO NASA-LEWIS

<u>I.D.</u>	<u>CONFIGURATION</u>
<u>THIN PANELS</u>	
CH-8	A1-C-B-C-A1
DL-18	A-B(45)-2B(-45)-B(45)-A
CC-36	A1-B-A2-B-A2-B-A2-B-A1
CH-13	A-D-D(90)-A
CC-11	A-3B-A
DL-27	2A1-B-A2-B-2A1
CC-18	2A1-B-2A2-B-2A1
CC-34	A-4B-A
<u>THICK PANELS</u>	
CD-6	6A-10B-4A-10B-6A
CH-10	8A-16D-8A
CH-5	2A-17C-2A
CD-5	8A-20B-8A
CD-24	5A-B-A-B-A-8B-2A-8B-A-B-A-B-5A
CH-16	4A-D-2A-D-A-7D-2A-7D-A-D-2A-D-4A
CD-21	4A-B-2A-B-A-B-A-14B-A-B-A-B-2A-B-4A
DL-9	5A-8B-B(45)-B(-45)-2B-B(-45)-B(45)- 2A-B(45)-B(-45)-2B-B(-45)-B(45)-8B-5A